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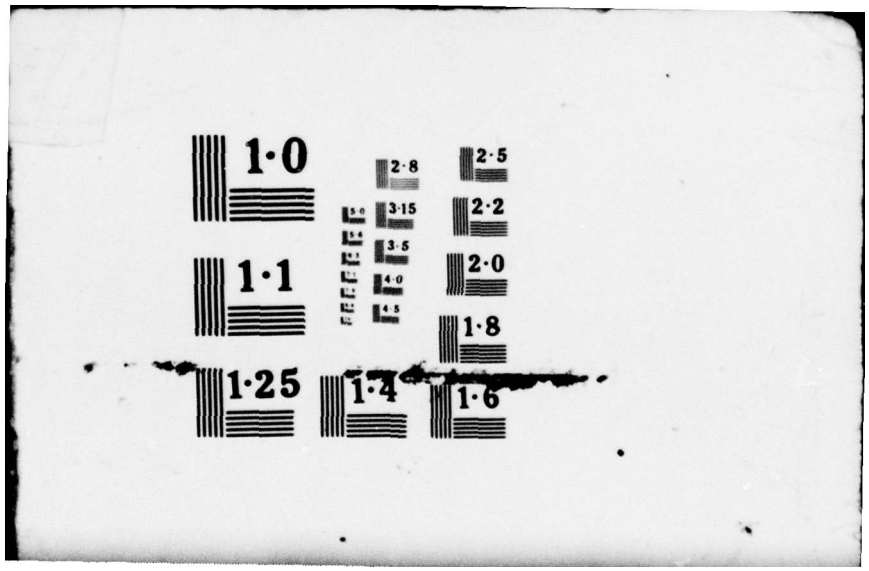
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AFAPL-TR-79-2037

MECHANISM OF TURBINE ENGINE LUBRICANT DEPOSITION

J. P. Cuellar, Jr.

Southwest Research Institute
San Antonio, Texas 78284

May 1979

Final Report March 1978-February 1979

AIR FORCE AERO PROPULSION LABORATORY
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Leon J. DeBrohun
LEON J. DEBROHUN
Project Engineer

H A Beane III, Acting
HOWARD F. JONES, Chief
Lubrication Branch

FOR THE COMMANDER

B. C. DUNNAM
B. C. DUNNAM, Chief
Fuels and Lubrication Division
Air Force Aero Propulsion Laboratory

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An investigation of the degradation and deposition mechanisms for inhibited polyol ester and diester lubricant basestocks is described. Basestocks trimethylolpropane triheptanoate and di(2-ethylhexyl) adipate containing antioxidants p, p'-dioctyldiphenylamine and/or phenyl-α-naphtylamine were studied in a dynamic thin-film condition with a moist-air atmosphere. Experiments were conducted with a device known as the rotating

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cylinder rig for the formation of controlled-thickness thin films (0.004 in.) at temperatures in the range of 350°F to 550°F. Analytical techniques for detection of lubricant or additive consumption and degradation products included gas liquid chromatography, principally, and gas chromatography/mass spectroscopy. Experiments showed that the antioxidants effectively retarded oxidation and deposition for both ester basestocks up to the time of complete additive consumption. A beneficial synergistic effect on deposition was noted at one concentration using the antioxidants in combination. Findings strongly suggest that an interaction between the ester oxidation products and system wearmetals is a significant factor in the deposition mechanism for both ester types.

PREFACE

This technical report was prepared by the Mobile Energy Division of Southwest Research Institute (SwRI). The effort was sponsored by the Air Force Aero Propulsion Laboratory (AFAPL), Air Force Systems Command, Wright-Patterson AFB, Ohio, under Contract No. F33615-76-C-2020 for the period 31 March 1978 to 2 February 1979. The work herein was accomplished under Project 3048, Task 304806, Work Unit No. 30480687, Mechanism of Turbine Engine Lubricant Deposition, with Messrs. L. J. DeBrohun, H. A. Smith, and P. W. Centers, AFAPL/SFL, as Project Engineers. Mr. J. P. Cuellar, Jr. of Southwest Research Institute was technically responsible for the work. The technical contributions of Dr. G. E. Fodor, Mr. F. M. Newman, Dr. C. P. Nulton, Mr. C. F. Rodriguez, and Mr. L. L. Stavinoha of Southwest Research Institute are acknowledged.

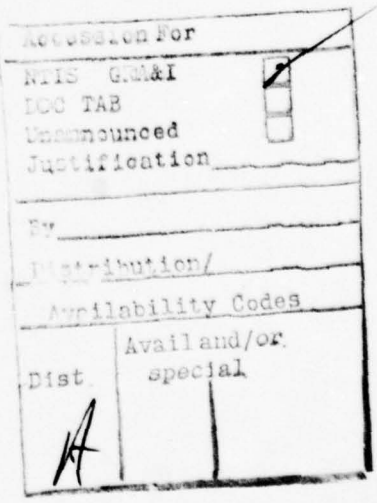


TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

SECTION I

INTRODUCTION

Commercially synthesized organic esters serve as the basestock for formulated lubricants currently used for main engine lubrication in virtually all gas turbine powered aircraft, both military and commercial. Two ester classes are predominant in such lubricant formulations: (1) dibasic acid esters formed via esterification of dibasic fatty acids and monohydric alcohols, and (2) neopentyl polyol esters of monobasic fatty acids and polyhydric alcohols. Frequently, to enhance rheological properties, blends of various ester types are employed. In the case of the polyol esters particularly, mixtures of esterification acids of varying chain length may also be used for the same purpose. Both ester classes utilize selected additives to achieve improved performance in properties such as oxidation stability, sludge dispersancy, foaming, and load-carrying capacity.

After some 30 years of use, study, and continuing improvement, it is understandable that turbine engine aircraft lubricants in present service generally provide satisfactory and reliable performance. Upgrading of lubricant formulations, lubricant specifications, and engine designs has been responsible for achievement of this performance level. Opposing this improvement trend has been the rise in engine operating temperatures due to increased aircraft speeds or, in recent years, increased inlet air temperatures to boost engine efficiency. Thus, research to define and evaluate lubricant performance properties continues. Investigation concerns the suitability of present fluids for current or future engine designs, as well as the promise of newly introduced lubricant formulations.

Of the numerous performance requirements which must be met by a lubricant formulation, deposition tendency is perhaps the most critical. The deleterious effect of lubricant deposits formed in the aircraft engine is manifested by plugging of lubricant jets and filters; malfunctioning of pumps, seals, and bearings; and accompanying increases in maintenance costs. Deposits result from thermal and/or oxidative breakdown of the lubricant formulations and are formed, primarily, in high-temperature engine zones which receive only indirect lubricant wetting, i.e., areas subjected to thin lubricant films.

The overall objective of this investigation was to examine and define the mechanisms involved in the process of deposition by ester-base lubricants. Experimental work utilized a rotating cylinder device, subsequently described in detail, for the formation of thin (0.004 in.) lubricant films under conditions of controlled temperature and atmosphere. The program schedule called for studies of a selected polyol ester basestock and a diester basestock, both with and without additives. Two interim reports on this work have been issued describing research on an uninhibited polyol ester,[1]* trimethylolpropane triheptanoate, and an uninhibited diester,[2]

* Superscript numbers in parenthesis refer to the List of References.

di(2-ethylhexyl) adipate. Degradation/deposition experiments with these basestocks included studies of thermal (inert atmosphere) and oxidative (air atmosphere) stability, using both dry and moist atmospheres for each type of environment. This final report presents the results of work employing the two ester basestocks blended with typical oxidation inhibitors--chemical compounds phenyl-α-naphthylamine (PANA) and p, p'-dioctyldiphenylamine (DOPA). All studies with the inhibited fluids were conducted with a moist air atmosphere.

Various chemical analyses were employed throughout this program to quantitatively, and, where possible, qualitatively monitor the reactants and products involved in the degradation process. The principal analytical techniques used for this purpose were gas chromatography (GC) and combined gas chromatography/mass spectroscopy (GC/MS).

SECTION II

ROTATING CYLINDER DEPOSITION TEST RIG AND PROCEDURES

Test Equipment

The device used in conducting oxidative stability experiments is known as the rotating cylinder deposition test rig. Details of the background and development of the device were originally described in AFAPL-TR-75-37.[3]

A schematic of the test rig is shown in Figure 1, with an identifying parts list given in Table 1. To provide for firm mounting and alignment, the assembly is installed on a lathe bed, item 1. The cylinder, item 17, is of stainless steel (Type 304) construction, open at one end and closed at the opposite end by a hemispherical internal surface connected to the drive shaft, item 20. Nominal cylinder dimensions are 4-in. ID with a horizontal surface of 4-1/2 in. At its open end, the cylinder is sealed by means of a carbon-face bellows seal, item 15, and its housing, item 14.

Passing through the seal housing are the oil-in line, item 5, and three bare-wire thermocouples, item 6, which can be positioned in contact with the fluid film on the cylinder ID. To provide hermetic sealing and lateral movement, the thermocouple probes enter the cylinder section though small metal bellows, item 12. All three thermocouples are mounted on micrometer spindles, item 9, to allow for accurate positioning and referencing. Viewed from the cylinder open end, the thermocouple at 0° provides input to a West temperature controller which activates a pair of 8-in. clam-shell heaters (not shown) encircling the cylinder, seal housing, and exposed portion of the cylinder drive shaft. The thermocouple at 120° is used to measure film thickness. Initial film contact by this sensor is indicated by an abrupt temperature rise measured by a potentiometer. Continued insertion to contact with the cylinder wall is evidenced by an electrical resistance measurement between the thermocouple wire and the OD of the cylinder. The thermocouple sensor at the 240° position is used for precise indication of the film temperature. This sensor is a commercial microminiature element with a bead diameter of slightly less than 2 mils. Readout of this thermocouple is by electronic digital indicator.

The cylinder rig lubricant and atmosphere flow systems are illustrated in Figure 2. Both systems are sealed from the environment between the gas-in (air or N_2) and exhaust points. The unheated lubricant reservoir is of borosilicate glass, with provision for magnetic-bar stirring of the fluid. The lubricant-in pump is a Zenith precision gear pump driven by a variable speed motor, with a 10:1 speed reducer to permit stable operation. The lubricant-in line includes a 200-mesh screen filter and an electrically heated preheater coil just before the line enters the cylinder. Preheating of the test lubricant to match the target film temperature is necessary to avoid discontinuities of both film temperature and thickness on the cylinder wall. The lubricant-in temperature is monitored by a bare-wire

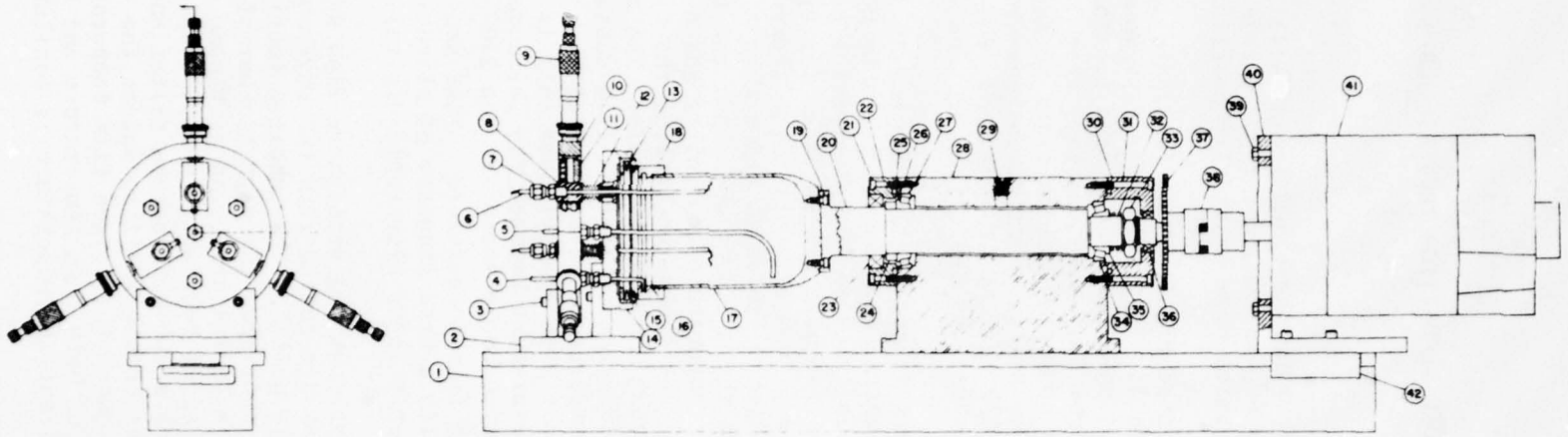


FIGURE 1. SCHEMATIC OF ROTATING CYLINDER RIG

TABLE 1. ROTATING CYLINDER
DEPOSITION RIG—PARTS LIST

Find No.	Part Name	Req'd
1	Lathe bed, Sears L9-1, Model No. 101,21400	1
2	Ring and seal bracket	1
3	1/4-20 x 3/4 hexagon socket cap screw	16
4	Scavenge oil line	1
5	Oil-in line	1
6	Thermocouple probe	3
7	Connector, Gyrolok 2 CMT-2-316	3
8	Connector adapter, SwRI A-3314-13	3
9	Micrometer, Starrett T262L-1	3
10	Micrometer ring, SwRI C-3314-15	1
11	Micrometer adapter, SwRI B-3314-16	3
12	Bellows, Metal Bellows Co. No. 60010-1	3
13	Bellows adapter, SwRI A-3314-18	3
14	Seal housing, SwRI C-3314-6	1
15	Seal, Sealol Inc. No. EJS-102573	1
16	Aluminum wire seal	1
17	Cylinder, SwRI B-3314-1	1
18	Seal counterface, SwRI B-3314-4	1
19	10-32 x 1/2 hexagon socket cap screw	4
20	Shaft, SwRI C-3314-39	1
21	10-24 x 1 hexagon socket cap screw	4
22	Front seal housing, SwRI B-3314-41	1
23	Seal, National No. 450194	1
24	Spacer, SwRI B-3314-42	1
25	O-ring, National No. 623008	2
26	Bearing cone assembly, Timken No. 13889	1
27	Bearing cup, Timken No. 13830	1
28	Bearing housing, SwRI C-3314-38	1
29	1/4-in. pipe plug	1
30	Rear seal housing, SwRI B-3314-40	1
31	10-24 x 1-3/4 hexagon socket cap screw	4
32	Spring	1
33	1-14 jam nut	1
34	Bearing cup, Timken No. 07196	1
35	Bearing cone assembly, Timken No. 07100	1
36	Seal, National No. 450567	1
37	Gear, 60-teeth	1
38	Coupling, Boston Gear No. FCR-FCBB-15	1
39	3/8-16 x 1 hexagon head cap screw	4
40	Motor bracket, SwRI D-3314-30	1
41	Boston Gear, 1/3 HP DC shunt motor	2
42	Motor bracket clamp	2

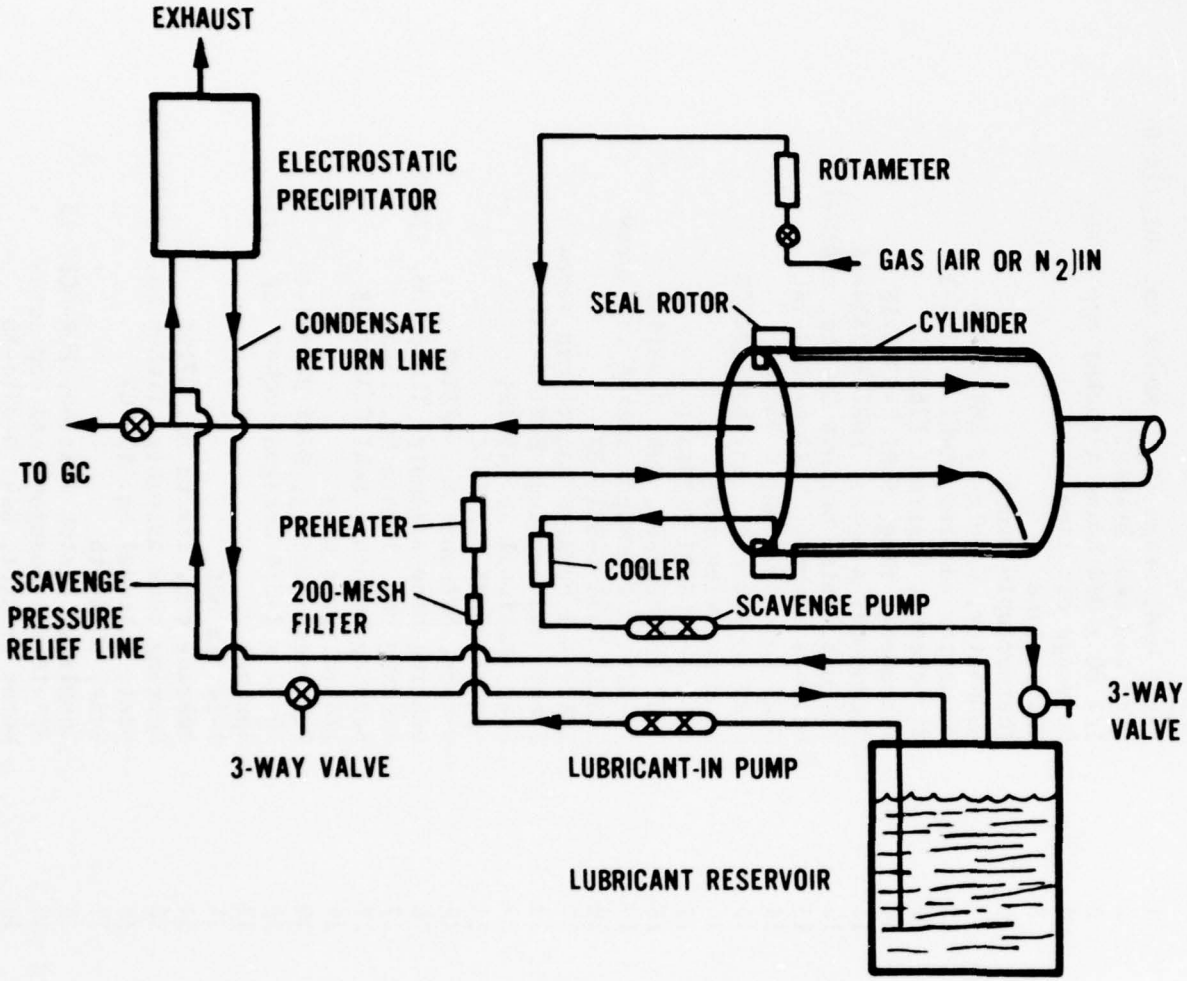


FIGURE 2. ROTATING CYLINDER RIG LUBRICANT/ATMOSPHERE FLOW SYSTEMS

thermocouple within the cylinder at the exit point of the oil-in line. The test lubricant is scavenged from a groove in the carbon seal counterface, item 18 (Fig. 1). The scavenge line passes immediately through a water-cooled heat exchanger (Fig. 2) to quench the degradation process. The scavenge pump, also a Zenith gear type, directs the fluid through a three-way sampling valve to the reservoir. Since over-scavenging is necessary, scavenged gas flows from the reservoir via a pressure relief line.

Gas (air or N_2) flow to the cylinder is metered through a line which exits near the closed end of the cylinder. The effluent gas line may be sampled for gas chromatographic (GC) analysis of oxygen content. Downstream of this point, the effluent line from the cylinder and the scavenge pressure relief line merge and flow to an electrostatic precipitator for recovery of condensable vapors. By gravity feed, the condensate is continuously returned to the lubricant reservoir.

The precipitator is composed of a 12-in. section of 2-in. pipe with an insulated precipitator wire centrally mounted within the pipe. In operation, the pipe section is grounded and 9000 volts of negative polarity applied to the wire. The entire device is encased by a short section of 8-in. pipe to contain debris in the unlikely event of a detonation within the precipitator.

At the precipitator operating volgage, there was some concern that, under oxidative conditions, a corona discharge capable of ozone generation might occur. Since ozone is highly reactive, oxidative attack of lubricant vapors within the precipitator might occur. However, atmosphere sampling just inside the precipitator exhaust line by means of a highly sensitive ozone detector (0.01 ppm) showed no evidence of the gas.

An overall view of the cylinder rig installation is seen in Figure 3. The gas chromatograph for oxygen analysis of the rig effluent gas is located at the far left. The cylinder, covered with blanket insulation, and the cylinder drive motor are mounted on the table near the photograph left center. The high-voltage precipitator is seen to the left and rear of the cylinder. Test lubricant pumps and the lubricant reservoir are mounted beneath the rig table. The rig instrumentation cabinet is at the center. The bench at the far right supports a strip-chart recorder and digital integrator on the lower shelf for the recording and processing output from the oxygen analysis GC. The GC instrument at the far right is used for the analysis of lubricant samples. The output from this GC is processed by a computer controlled data system subsequently described. Some of the components of this system seen in Figure 3 are the terminal teletype, the analog/digital (A/D) converter to the right of the teletype, and the acoustical coupler and telephone below the strip-chart recorder.

Test Procedures and Conditions

Cylinder rig tests reported here in were conducted in multiples of 5 hr of test time. Normally, runs were performed with two-shift operation

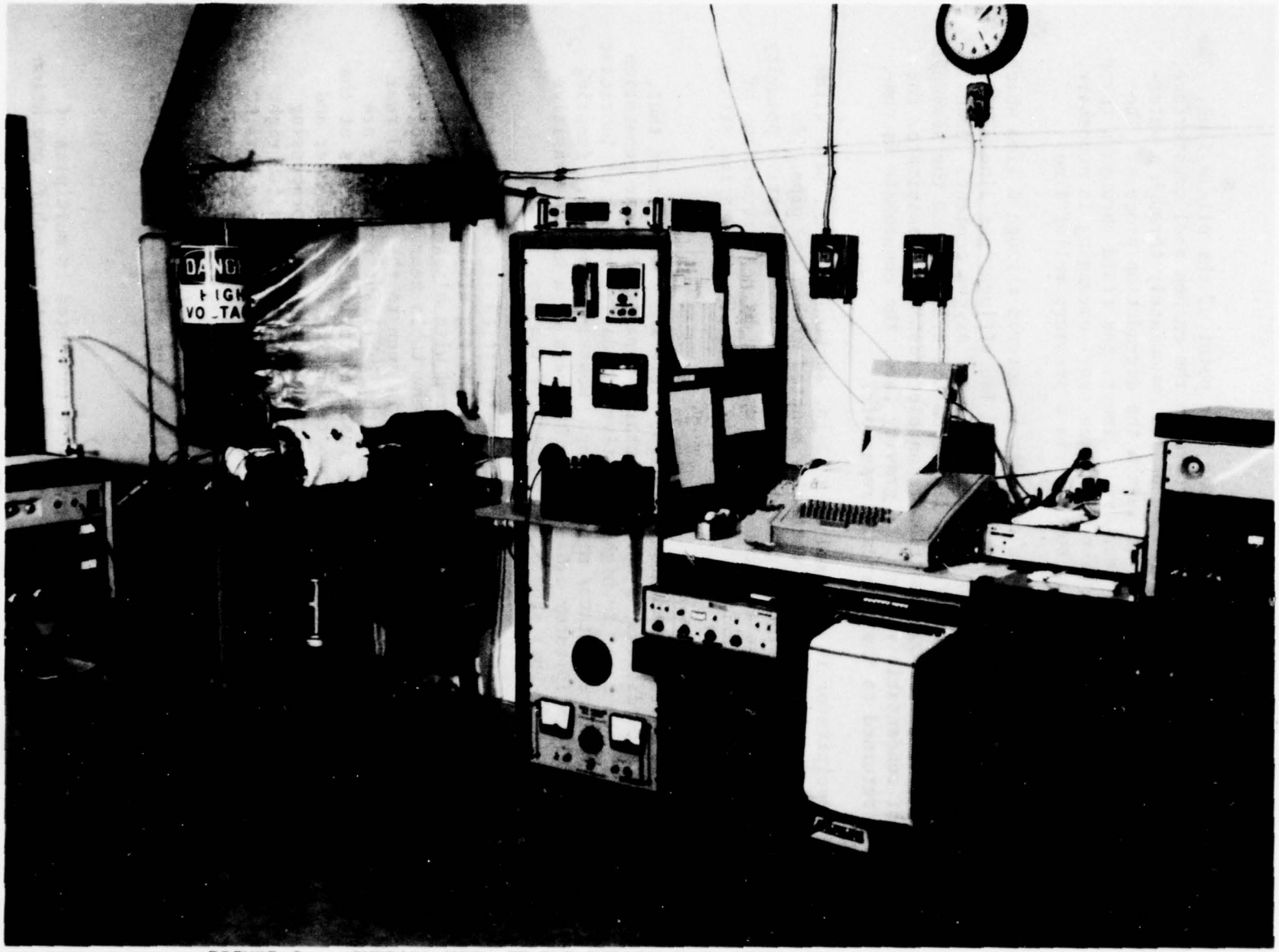


FIGURE 3. OVERALL VIEW OF THE ROTATING CYLINDER RIG AND INSTRUMENTATION

to obtain 15 hr of continuous run time per day. During a run, verification and recording of flow rates, temperatures, and film thickness were performed at 30-min intervals. Rotational speed of the cylinder was also recorded at this time, but speed adjustments were made, as required, to maintain the desired film thickness. Test lubricant samples (10 cm^3) were taken at 5-hr intervals. A sample of the condensate from the electrostatic precipitator was normally taken at the termination of each test. GC analysis of the cylinder effluent atmosphere was made each 30 min.

Certain test conditions, with values selected on the basis of prior work,[3] were held constant throughout the program. These were:

Film thickness	0.004 in.
Lubricant flow	10 cm^3/min
Airflow	100 ℓ/hr
Lubricant charge	1000 cm^3

Test duration was generally dictated by the extent of lubricant deterioration. That is, runs were terminated if excessive increases in sample viscosity and/or neutralization number occurred. No lubricant makeup for losses or intermediate samples was made during the tests.

Film temperature was the only variable test condition for runs reported herein. This condition was adjusted in increments of 25°F to encompass, in general, the temperature capability of the test lubricant. All tests utilized a moist air atmosphere, with water content controlled at 10 $\pm$ 1 mg per liter of air.

In addition to the specification of test conditions of film temperature, film thickness, and total oil flow rate, calculations were made relative to average and total lubricant residence times. The average residence time was a calculated value for a "single pass" based on the average velocity of the lubricant, which in turn was based on lubricant flow rate and the cross-sectional area of the film. Thus, the following relationships apply:

$$t = \ell / v \qquad (1)$$

$$v = Q / c \qquad (2)$$

$$c = \pi/4 [d^2 - (d - h)^2]$$

where t = average residence time, sec

ℓ = cylinder length, cm

v = average film velocity, cm/sec

Q = lubricant flow rate, cm^3/sec, corrected for thermal expansion

c = film cross section, cm^2

d = cylinder ID, cm

h = film thickness, cm

It is noted that the above relations are simplified in that boundary effects and, possibly, rotational effects are not considered. If one considers a unit volume of lubricant, the volume will be exposed to the high-temperature cylinder surface in one cycle for the duration of the average residence time, t. If it is assumed that each fluid increment has an equal probability of undergoing the same number of cycles during test, a total residence time may be calculated as follows:

$$E = \frac{TQt}{V} \qquad (3)$$

where Q and t are as above and

E = total residence time, sec

T = test duration, sec

V = lubricant charge, cm^3

To account for fluid losses during a test, Eq. (3) was summed for each 5-hr test period using a mean volume indicated by oil loss measurement, and correcting for intermediate lubricant sample withdrawals. Under these circumstances, it is observed that the total residence time per 5-hr period increases with test time. Similarly, unusually high oil losses (e.g., due to improper carbon-seal performance of a temporary nature) increase the total residence time, especially if the losses occur during the early hours of the test.

Deposit Rating Procedure

The deposit rating procedure used to describe numerically the deposits occurring within the cylinder was essentially that used in the 48-hr bearing deposition test[4] except that only one surface was inspected, viz., the surface of the cylinder ID.

A demerit rating number was selected to identify the different types and thicknesses of deposits present. Demerit values range from 0 to 20, defined as follows:

Deposit Type	Demerit Rating Number		
	Light	Medium	Heavy
Varnish	1	3	5
Sludge	6	7	8
Smooth carbon	9	10	11
Crinkled carbon	12	13	14
Blistered carbon	15	16	17
Flaked carbon	18	19	20

This demerit number was multiplied by a number from 0 to 10, corresponding to the percent of the area, 0 to 100 percent, covered by that deposit type. In the event that more than one type of deposit was present on the rated area, the deposit rating was then the total of the individual rating values necessary to account for 100 percent of the rated area. In any event, double ratings, such as sludge over varnish, were not used. The deposit rated was that which was visible without the removal of another deposit, except in the case of sludge over carbon. In such instances, the more severe deposit type was used in the rating calculations.

SECTION III

LUBRICANT ANALYSIS PROCEDURES

All intermediate and final lubricant samples taken during the cylinder rig tests were routinely analyzed for kinematic viscosity by ASTM Method D 445 and for neutralization number (total acid number) by ASTM Method D 664.

GC was the principal tool used in following changes in the major ester component of the lubricant basestock, as well as other significant constituents in the new or used samples. The analysis was performed with an instrument equipped with a hydrogen flame ionization detector (FID). The conditions and column materials used with the procedure are summarized here:

Liquid phase	OV-17, 2%
Solid phase	Gas Chrom Q, 60/80
Column length	17 ft
Column tubing	0.125-in. OD x 0.093-in. ID S.S.
Column efficiency	1200 theor. plates
Oven temperature	
Initial	180°-210°C
Final	300°-320°C
Program rate	8°C/min
Injector temperature	300°-320°C
Detector temperature	320°-340°C
Carrier gas (He) flow	25 cm^3/min
Sample size	0.2 μℓ
Internal standard	n-tridecane, 10 wt %
Injection procedure	On-column

The GC output signal was processed by a Hewlett-Packard 3354A laboratory data system available at SwRI. In this system, the A/D converter digitizes the GC signal and transmits the data via a cable loop (serving several SwRI laboratories) to a general purpose computer. Analytical results from the computer and report print-outs are received by the terminal teletype over a direct-wired telephone line, with associated acoustical couplers at each end of the line. The availability of this system to the program significantly benefitted the effort with regard to the accuracy and speed of GC data processing.

Because of the unavailability of suitable standards and, in some cases, of full knowledge of chemical structure, a common GC response factor relative to n-C_{13} was used for all lubricant components. It should also be noted that component concentrations for degraded samples, reported as weight percent, are subject to some error since no attempt was made to achieve total recovery of gaseous degradation products such as moisture, CO, or CO_2. However, it is believed that the error introduced by the lack of a material balance determination would not be appreciable.

Selected lubricant and deposit samples were also analyzed by various other methods in an effort to characterize the chemical structure of these materials. Analytical techniques utilized for this purpose included infrared spectrophotometry, X-ray fluorescence spectrometry, mass spectrometry, and combined gas chromatography/mass spectrometry (GC/MS). Analyses by GC/MS were performed with a Finnigan Model 3300F instrument interfaced with a computer for both instrument control and data acquisition and analysis. The GC procedures and conditions used with the GC/MS instrument were similar to those previously described for GC analysis of lubricant samples. The GC/MS instrument may be operated by use of an electron impact ionization source (70 electron volts) or a chemical ionization source (methane reagent gas), each equipped with a quadrupole mass filter. The GC/MS facility has access to the Cyphernetics spectra library known as the mass spectral search system (MSSS).

Selection of the analytical procedures used in this study was based principally on prior knowledge of the field and the finding of the literature search presented in a previous report[1] on this program.

SECTION IV

TEST MATERIALS

A single batch each of a trimethylolpropane triheptanoate (TMP) ester, coded O-76-5, and a di(2-ethylhexyl) adipate (DEHA) ester, coded O-77-1, were provided by AFAPL for use in this study. These basestocks were commercially available materials containing, as received, no additives. The following basic properties were determined for the two fluids:

	DEHA	TMP
100°F Vis, cs	8.19	15.23
210°F Vis, cs	2.37	3.53
Neut. No., mg KOH/g	0.03	0.02
Gravity, °API/60°F	21.0	15.2

Extensive chemical characterization of the TMP basestock was described in reference 1. Analysis for organic acids and alcohols, in addition to GC/MS studies, resulted in a compositional breakdown for the material as shown in Table 2. Numerics in this table used to identify the primary compounds refer to the acyl group carbon chain length. Peak 7 was identified as a pentaerythritol (PE) ester, with a C_7 acyl group at each of the four possible acyl positions.

Similar characterization work[2] with the diester showed a DEHA content of 99.7 wt percent. Two contaminants identified in the basestock at less than 0.2 wt percent each were the DEHA parent alcohol, 2-ethyl-1-hexanol, and a diester similar to DEHA but with some slight variation in one of the alcohol groups.

Table 3 summarizes the lubricant codes and additive concentrations used for blends with the antioxidants p, p'-dioctyldiphenylamine (DOPA) and phenyl-α-naphthylamine (PANA). The DOPA additive was investigated in both basestocks at two percent concentration and, similarly, PANA at one percent. Utilizing the TMP basestock only, combinations of the inhibitors were investigated at a total additive concentration of two or one percent, while maintaining a 2:1 ratio of DOPA:PANA. Thus, for example, the two percent additive package in lubricant Y-1011 consisted of two parts DOPA and one part PANA.

TABLE 2. O-76-5 BASESTOCK CHARACTERIZATION FOR MAJOR COMPONENTS

Peak	Concentration by GC, wt %	Primary Compound
1	0.7*	TMP diester-aldehyde
1a	0.2*	TMP diester-alcohol
2	2.9	TMP-477
3	0.6	TMP-577
4	2.5	TMP-677
5	89.9	TMP-777
6	0.7	TMP-778
7	0.9	PE-7777

* Approximation.

TABLE 3. TEST LUBRICANTS

Code	Basestock	Additive
O-77-1	DEHA	None
Y-1004	DEHA	2% DOPA
Y-1007	DEHA	1% PANA
O-76-5	TMP	None
Y-1006	TMP	2% DOPA
Y-1010	TMP	1% PANA
Y-1011	TMP	2% (DOPA, PANA)*
Z-1000	TMP	1% (DOPA, PANA)*

* Additive ratio 2:1 of DOPA:PANA.

SECTION V

SUMMARY OF PRIOR WORK

As previously noted, two interim reports have been published on this program describing thermal and oxidative stability studies with the uninhibited TMP[1] and DEHA[2] ester basestocks. For the purpose of continuity, this section will review the findings presented in those reports.

TMP Basestock

With an inert (nitrogen) atmosphere, thermal degradation of the TMP triheptanoate basestock was slight at 600°F film temperature. Cylinder rig tests at 650°F showed increased ester breakdown, especially with a moist atmosphere. For this condition, the principal degradation products were diesters, heavy constituents indicated by GC residue, and, as found primarily in condensate samples, heptanoic acid. Only varnish-type deposits were formed in the thermal stability test series. Chemical analysis of the deposit of a varnish appearance recovered from one run showed the composition of the material was primarily inorganic; however, the quantity of the deposit available for analysis was extremely small.

Employing film temperatures of 350° and 400°F, oxidative deterioration of the polyol ester was significant. Use of a moist air atmosphere did not accelerate degradation, probably because the intensity of the oxidative attack was so severe that any hydrolytic effect was obscured. Aside from quantitative differences, the products of ester oxidation were similar to those found in the thermal stability experiments, i.e., compounds postulated to be C_7 diesters, GC residue components, and heptanoic acid. In addition, the oxidation experiments showed the apparent formation of triesters in the gas chromatogram region associated with the presence of C_8 acyl groups. A possible explanation for this phenomenon might be associated with a recombination of free C_8 acid and partially hydrolyzed esters.

On the basis of average oxygen consumption rate data, calculated energies of activation for the polyol ester were 9.9 Kcal/mole with moisture present, and 11.8 Kcal/mole for dry air. The fact that these values were considerably lower than expected was thought to be the result of the "rig dependence" of data obtained.

Elemental analysis of various sludge deposits from the TMP oxidation experiments indicated a high inorganic content, primarily iron and other metals. Mass spectrometry confirmed the inorganic nature of the deposit samples and also showed the presence of the C_7 acyl ion (m/e 113). Analysis of fluid samples taken for one test revealed a rapid buildup in iron contents in the later hours of the test, with the highest iron concentration found in the acidic condensate sample. Microscopic examination and X-ray fluorescence "fingerprinting" of the deposit and suspended matter in the test fluid indicated the materials were of similar morphology and metal content.

The foregoing findings suggested that, under the conditions investigated, the mechanism of deposit formation by the polyol ester was through a corrosive wear process. Assuming the cylinder rig apparatus was inherently a wear-free device, cylinder wall deposits were postulated to be metallo-organic compounds resulting from attack of system metals by the acidic constituents generated, primarily heptanoic acid. However, this theory regarding the source of metal could not be confirmed by subsequent ferrographic analysis of fluid samples performed at AFAPL. Analysis of oxidative test samples from two runs in which significant light sludge deposits were formed showed negligible corrosive wear particles in one case, and none in the other. Particles were primarily of the normal and severe wear type, fatigue chunks, and oxides. Nevertheless, XRF data for deposit samples from both tests indicated, in addition to iron, an appreciable amount of nickel (0.2 percent) present for the test in which a few corrosive wear particles were ferrographically determined. This metal is present in the composition of several components of the cylinder rig, including the cylinder. Nickel is not found, however, in any of the components in rubbing contact, namely, the lubricant pump gears or body, or the seal counterface.

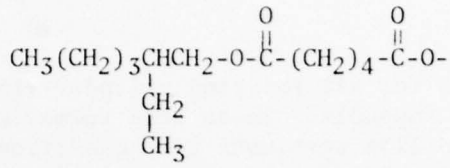
DEHA Basestock

Thin-film thermal stability experiments with an uninhibited lubricant basestock, di(2-ethylhexyl) adipate, demonstrated a significant performance effect for moisture. The effect was beneficial with respect to the degree of ester attack and deposit formation. The thermal degradation products of the diester included three low-boiling compounds characteristic of the parent alcohol of the compound, 2-ethyl-1-hexene, 2-ethyl-1-hexanol, and 2-ethyl-1-hexanoic acid. Atmospheric moisture apparently promoted formation of the acid. Molecular still separations of end-of-test samples showed a much higher viscosity and neutralization number for the moist nitrogen test residue.

A major effect for moisture was shown by deposition results for the thermal stability test series. With moisture present, an innocuous light varnish deposit occurred. Using a dry nitrogen atmosphere, significant carbon-type deposits were formed. XRF analysis of this material showed appreciable metal contents, principally iron. Significant amounts of iron and chromium were also found in the test lubricant samples from the dry nitrogen experiment. Analysis of the carbon deposit by mass spectroscopy yielded a mass spectrum characteristic of the original diester.

The low boiling compounds resulting from thermal deterioration of the adipate ester suggest cleavage of the alcohol group. The fate of the remaining fragment of the molecule may be in the formation of condensed residue products or, in the case of the dry nitrogen tests, carbon deposits formed by metal combination compounds. A metal involvement in the deposition process seems certain. The mass spectrum of the deposit sample was virtually identical to that of the original diester. However, assuming a metal complex, the following organic moiety might exhibit a mass spectrum

indistinguishable from that of the diester:



Ferrographic results provided by AFAPL showed that nascent wear particles were present in the thermal stability test samples. XRF of fluid samples indicated negligible metal amounts for the moist nitrogen determination (light varhish deposit) and significant amounts for the dry nitrogen test (carbon deposits). One source of these particles is now thought to be the rotating counterface (item 18 of Fig. 1) of the cylinder carbon seal. Weight measurements of this component, constructed of AISI Type 440C stainless steel, indicated metal losses as high as 220 mg can occur. This amount would be sufficient to produce an iron content on the order of 400-500 ppm in the reduced test fluid volume at the end of a run.

With a wearmetal involvement in the deposition process, it is difficult to describe the precise role of moisture in the mechanism. Three distinct phenomena can be visualized: 1) moisture served to mitigate the wear tendency of the carbon-steel seal surfaces; 2) moisture promoted formation of a degradation product of the diester, possibly free adipic acid or 2-ethyl-1-hexanoic acid, which mitigated wear; and 3) moisture might in some way passivate the nascent wear particles to block any interaction with the diester products. The latter speculation would appear somewhat inconsistent with ferrograph data which indicated some deposit-coated metal particles present in the moist nitrogen test.

With an oxidative atmosphere, cylinder rig experiments with the diester basestock did not establish a conclusive effect for moisture. At a 350°F film temperature, the poor repeatability of three tests using dry air did not permit a distinction versus the degree of deterioration in duplicate determinations using moist air. Although the amount of reacted basestock was extensive, 35-50 percent, for both oxidative conditions, the deposition tendency of the diester was insignificant. Analytical work with fluid samples from a "representative" moist air test showed numerous oxidation products in evidence. In addition to the low-boiling compounds formed by thermal degradation, the oxidation test resulted in various mixed diesters and mono-esters of C_2, C_4, and C_5 acyl groups, as well as four compounds boiling just above the original diester.

SECTION VI

EXPERIMENTAL RESULTS AND DISCUSSION

General

Individual data sheets for all rotating cylinder rig tests referred to herein are included in the Appendix. These data summaries are arranged in order of test number and list pertinent test conditions, the cylinder rig deposit rating, and basic lubricant performance data. Oxygen consumption values are expressed in liters at operating temperature and pressure (OPT), corresponding to ambient atmospheric pressure and temperature (75° to 80°F). Data for the amount of unreacted ester remaining, determined by GC, refer to the primary ester component present in the basestock.

DEHA Results

A summary of cylinder rig test data for the neat (lubricant code O-77-1) and inhibited diester basestock is given in Table 4. It is apparent that both antioxidant types very effectively extended the oxidative stability of the fluid, with the PANA additive being superior to DOPA in film temperature capability by some 50°F. This observation is based on the extent of ester attack and additive remainder values (by GC analysis) listed in Table 4. For example, total consumption of the DOPA material was noted at 40 hr using a 475°F film temperature. Total PANA depletion occurred at 35 hr with a 525°F film temperature.

Aside from the difference in temperature capability, both of the inhibited lubricants exhibited similar mechanisms in oxidative degradation. Prior to the time of total additive depletion, ester consumption was slight and only moderate changes in lubricant viscosity and neutralization number were observed. During this period, GC analysis showed no products present other than high boiling materials (GC residue) not eluted by the normal procedure. These GC residue amounts accounted for essentially all of the consumed diester.

Following total depletion of the DOPA or PANA additive, accelerated lubricant deterioration occurred. Figure 4 shows the abrupt attack of the diester, coincident with additive loss. This figure also illustrates the induction period provided by the antioxidants, contrasted with the consistent ester consumption in the uninhibited fluid. Also corresponding to the time of additive depletion were accelerated increases in oxygen uptake, fluid viscosity, and neutralization number. Multiple degradation products were also formed, similar in type and distribution to those for the uninhibited DEHA.[2] These included products characteristic of the DEHA parent alcohol, such as 2-ethyl-1-hexene, 2-ethyl-1-hexanol, and 2-ethyl-1-hexanoic acid, as well as mixed diesters and the adipate mono-ester, 2-ethylhexyl hydrogen adipate. The latter half-acid ester was present in amounts of 1 to 1.5 wt percent for those inhibited DEHA tests indicating severe deterioration. As evident in Table 4, the GC residues constituted, by far, the largest amount of reaction products generated.

TABLE 4. SUMMARY DATA FOR NEAT AND INHIBITED DI(2-ETHYLHEXYL) ADIPATE

(Moist air)

Lubricant Code, Additive	Film Temp, °F	Test Time, hr	Total Res Time, sec	100°F Vis Incr, %	NN Change, mg KOH/g	Deposit Rating	Deposit Type	O_2 Consump, % at OPI	Unreacted Ester, wt%	Additive Rem, wt%	GC Res, wt %	Test No.
O-77-1, none	350	50	697	34	13.5	10	LV	21.6	66	--	23	44
	350	50	614	34	15.4	10	LV	23.4	65	--	22	45
	375	35	459	39	14.1	10	LV	25.1	65	--	19	53
	375	35	429	40	13.4	10	LV	28.0	66	--	22	54
Y-1004, 2% DOPA	350	135	2935	2	0.3	0	Clean	4.1	98	1.6	2	55
	400	100	1411	5	1.2	10	LV	6.3	94	0.7	6	56
	450	95	1814	129	35.5	10	LV	39.8	50	0.0 (80 hr)	40	57
	475	50	674	123	29.5	10	LV	56.0	49	0.0 (40 hr)	40	58
Y-1007, 1% PANA	450	100	1447	6	2.2	12	LV	7.4	96	<0.05	5	63
	475	100	1479	8	3.1	30	MV	9.1	94	<0.05	4	64
	500	100	1451	8	3.7	50	HV	7.4	94	0.08	5	65
	525	45	584	156	22.4	130	MCC	46.1	51	0.0 (35 hr)	40	67

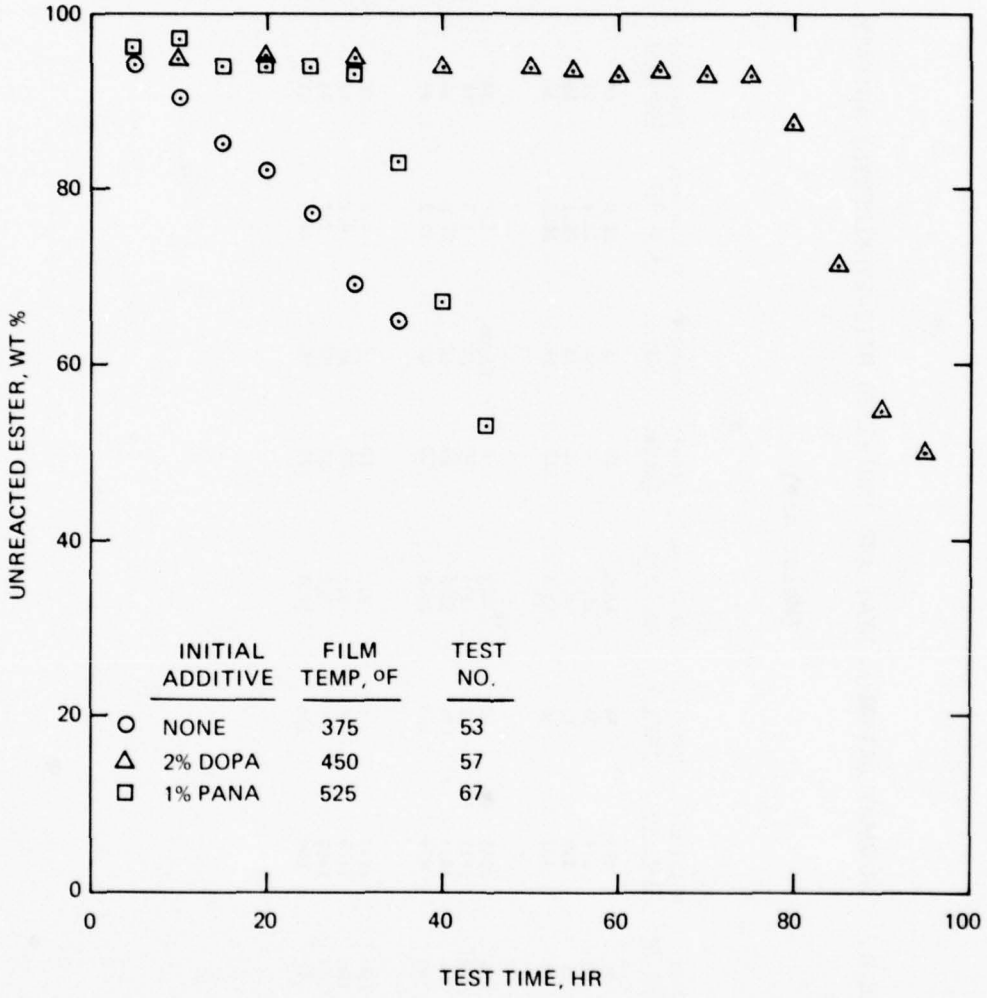


FIGURE 4. DEHA ESTER CONSUMPTION TRENDS

Figure 5 illustrates the rate of depletion of the two antioxidants in DEHA at 450°F film temperature. Semi-logarithmic plots of these data yield a straight line for both materials, characteristic of a first-order chemical reaction.

Contrary to findings reported for other types of deposition test devices, e.g., references 5 and 6, cylinder rig experiments listed in Table 4 did not indicate a deleterious effect for the antioxidants, at comparable temperatures, in regard to deposition tendency. At film temperatures even higher than those used with the neat basestock, only light varnish (LV) deposits were obtained with the DOPA additive present. A similar deposit rating was noted for the PANA-inhibited fluid at 450°F. Increasing film temperature with Y-1007 resulted in medium and heavy varnish deposits, while the highest temperature (525°F) produced a medium crinkled carbon (MCC) deposit in a 45-hr test period. Thus, relative to the DOPA inhibitor, PANA indicated some tendency for promotion of deposits at the higher film temperatures.

Earlier work[1,2] with the neat ester basestocks showed a distinct involvement for metals in the deposition process. Accordingly, XRF analyses were performed on lubricant and deposit samples from the highest film temperature test with each of the inhibitors in DEHA. The following results were obtained for these samples:

Test No. 58, DEHA + 2% DOPA		Test No. 67, DEHA + 1% PANA	
0-40 hr	Nil	0-35 hr	Nil
45	65 ppm Fe	40	40 ppm Fe
50	150 ppm Fe	45	445 ppm Fe
Condensate	325 ppm Fe	Condensate	<30 ppm Fe
Deposit	Insuff. sample	Deposit	4.2% Fe
			2.3% Si
			0.8% S

Referring to the deterioration trends for these two runs, it is seen that significant quantities of iron occurred in the lubricant samples immediately following complete additive depletion. It was not feasible to recover a reasonable sample amount of the light varnish deposit generated in Test No. 58. The crinkled carbon material obtained in Test No. 67 showed appreciable quantities of inorganics, principally iron. Silicon is present in some of the rotating components of the cylinder rig, but it is believed more likely that this element was a residual from the rig cleaning procedure. No source of sulphur in the system can be identified, except possibly as an impurity in the cleaning solvents.

Additional characterization of the Test No. 67 deposit material was performed by analysis for the basic elements carbon, hydrogen, nitrogen, and oxygen, and by mass spectroscopy. The following data for elemental composition

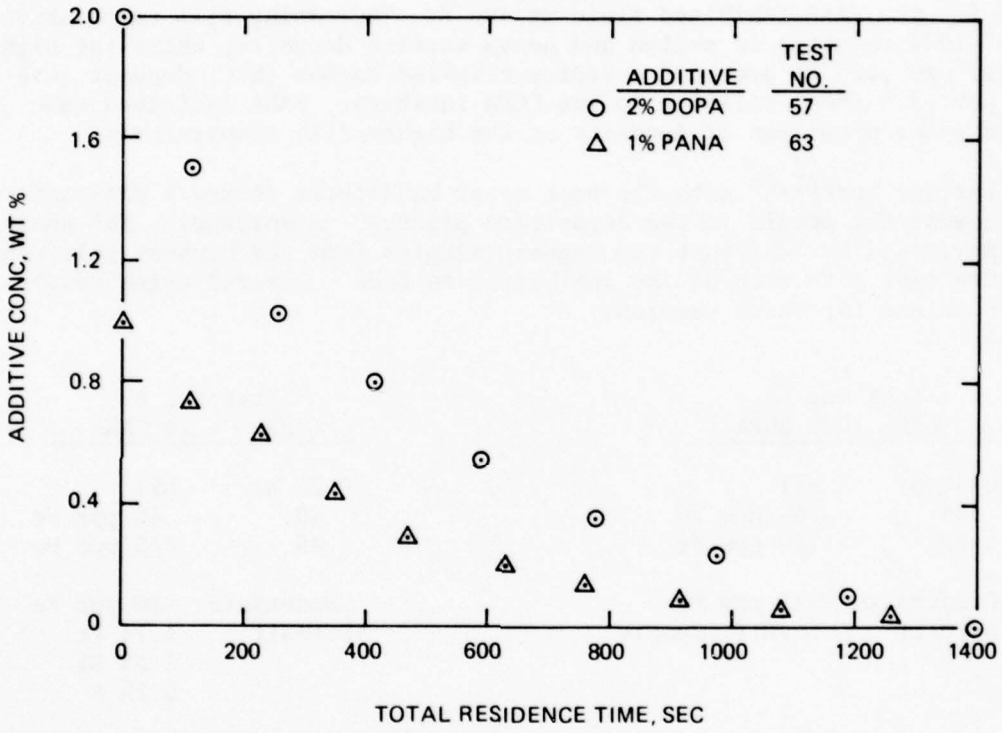


FIGURE 5. ADDITIVE DEPLETION IN DEHA AT 450°F FILM TEMPERATURE

are compared with theoretical values for the DEHA ester containing one percent PANA:

	Weight Percent	
	Theor.	Deposit
C	71.5	70.6
H	11.3	5.7
N	0.1	1.2
O	17.1	22.1

Relative to the original ester, the deposit sample indicated some increase in oxygen and a significant increase in the carbon/hydrogen ratio. The nitrogen analysis suggests some involvement of the amine inhibitor in the deposition process, at least during the exposure period prior to additive depletion.

The mass spectrum of the deposit sample, shown in Figure 6, was obtained by solid probe insertion of the material using electron impact ionization. The analysis was performed after repeated heptane extractions to remove residual fluid. The spectrum contains several ions characteristic of the DEHA ester,[2] although there was some reduction in the relative abundance of ions attributable to larger fragments of the DEHA molecule. The base peak at m/e 55 and high abundance ions at m/e 57, 70, 71, and 112 are derived from the ester alcohol group. The ions at m/e 129 and 147 are characteristic of the diester acyl group, whereas m/e 241 and 259 fragments represent ions resulting from loss of a single alcohol group. The latter two ions were of very low abundance, particularly if it is noted that the spectrum above m/e 140 utilizes a 3X intensity amplification (as indicated by the figure notation, >140 x 3). The mass spectrum does not contain any ions which could be ascribed to the PANA additive. The appearance or the increased abundances for ions m/e 27, 28, 41, and 42 may be attributed to the addition of a double bond in the alkyl fragments producing ions of the form $C_n H^+_{2n-1}$ and $C_n H^+_{2n}$ series. This indication of unsaturated aliphatic hydrocarbons is in agreement with the high carbon/hydrogen ratio shown by elemental analysis of the deposit.

TMP Results

Cylinder rig test data for oxidative deterioration of the polyol ester fluid are summarized in Table 5. As with the diester basestock, use of the antioxidants substantially increased the temperature capability of the polyol ester with respect to oxidative resistance.

When employed individually, the amine additives were effective to 500°F with respect to inhibition of ester attack and deposit formation. Cylinder deposits were nil with both additives at this temperature, whereas the neat basestock formed light sludge (LS) at much lower film temperatures. The mode of polyol ester attack was likewise similar to that for the inhibited diester as seen for typical data in Figure 7. Moderate and gradual

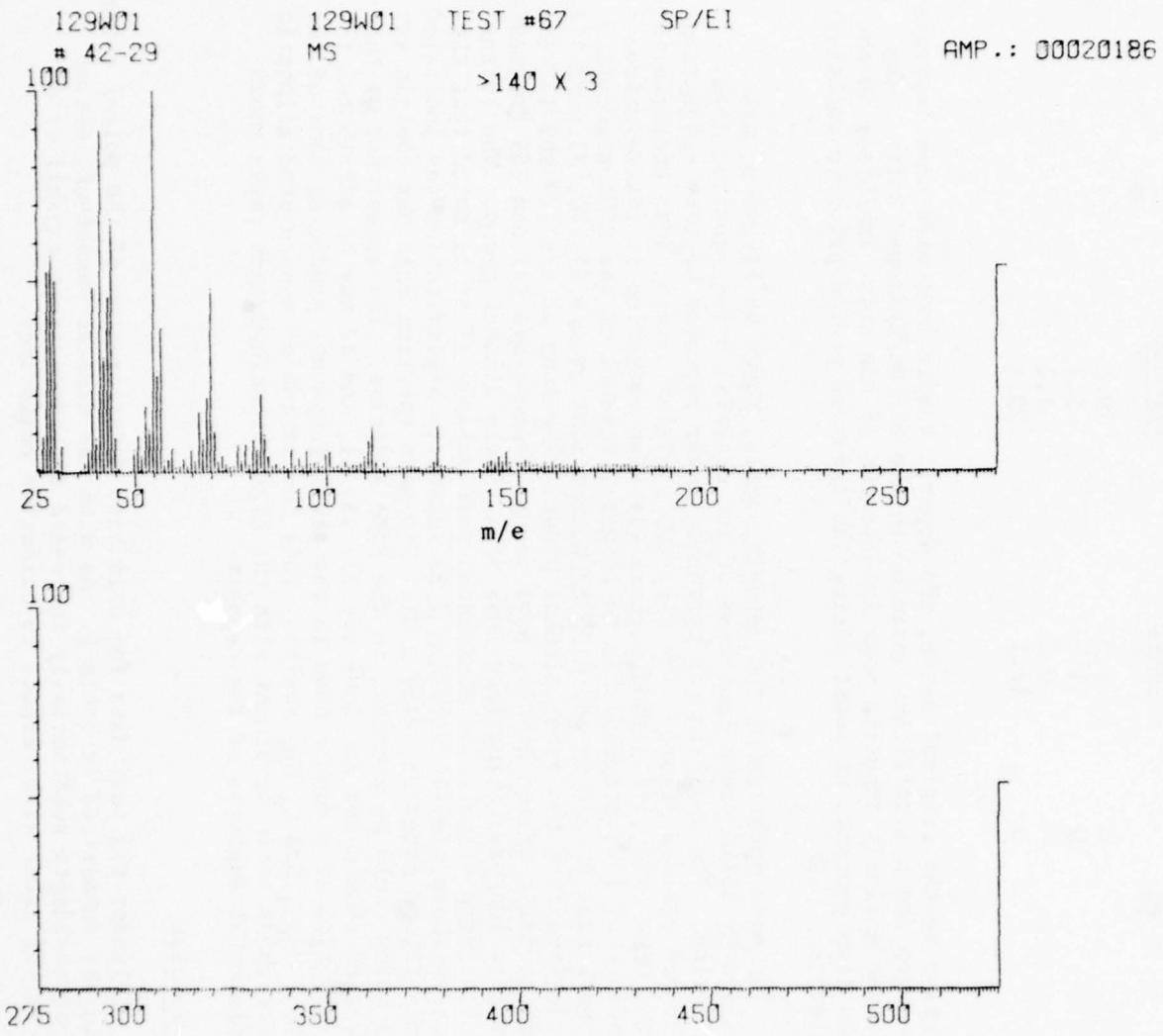


FIGURE 6. MASS SPECTRUM FOR TEST NO. 67 DEPOSIT SAMPLE

TABLE 5. SUMMARY DATA FOR NEAT AND INHIBITED TRIMETHYLOLPROPANE TRIHEPTANOATE

(Moist air)

Lubricant Code, Additive	Film Temp, °F	Test Time, hr	Total Res Time, sec	100°F Vis Incr, %	NN Change, mg KOH/g	Deposit Rating	Deposit Type	O_2 Consump, % at OPT	Unreacted Ester, wt %	Additive Rem, wt % DOPA	Additive Rem, wt % PANA	GC Res, wt %	Test No.
O-76-5, none	350	90	1693	68	11.6	60	LS	22.1	47	-	-	40	40
	350	100	1800	73	13.2	60	LS	26.6	44	-	-	42	43
	400	50	632	79	10.8	60	LS	39.3	44	-	-	43	41
	400	50	651	60	9.8	50	LS	33.3	47	-	-	40	42
Y-1006, 2% DOPA	450	100	1303	8	0.8	0	Clean	8.0	94	1.3	-	3	59
	500	100	1274	16	1.4	0	Clean	10.8	87	0.6	-	7	60
	525	70	871	312	14.8	60	LS	79.4	29	0.0	-	53	62
	550	25	280	188	16.0	60	LS	87.5	29	0.0	-	53	61
Y-1010, 1% PANA	500	100	1266	9	0.9	0	Clean	4.2	92	-	0.2	5	69
	525	97	1976	494	15.9	60	LS	19.1	31	-	0.0	53	70
	550	25	269	98	12.5	60	LS	62.6	40	-	0.0	40	71
Y-1011, 2% (DOPA, PANA)*	500	100	1530	12	1.0	0	Clean	5.6	93	0.6	0.02	6	72
	525	95	1191	72	20.2	105	Carbon	45.6	50	0.0	0.0	37	76
	550	25	279	89	12.9	90	LS/carbon	50.1	49	0.0	0.0	37	77
Z-1000, 1% (DOPA, PANA)*	525	45	515	100	14.1	10	LV	37.6	36	**	0.0	44	78
	525	25	312	114	14.9	10	LV	45.0	35	**	0.0	49	79

* Additive ratio 2:1 of DOPA: PANA.

** Indeterminable due to GC peak interference.

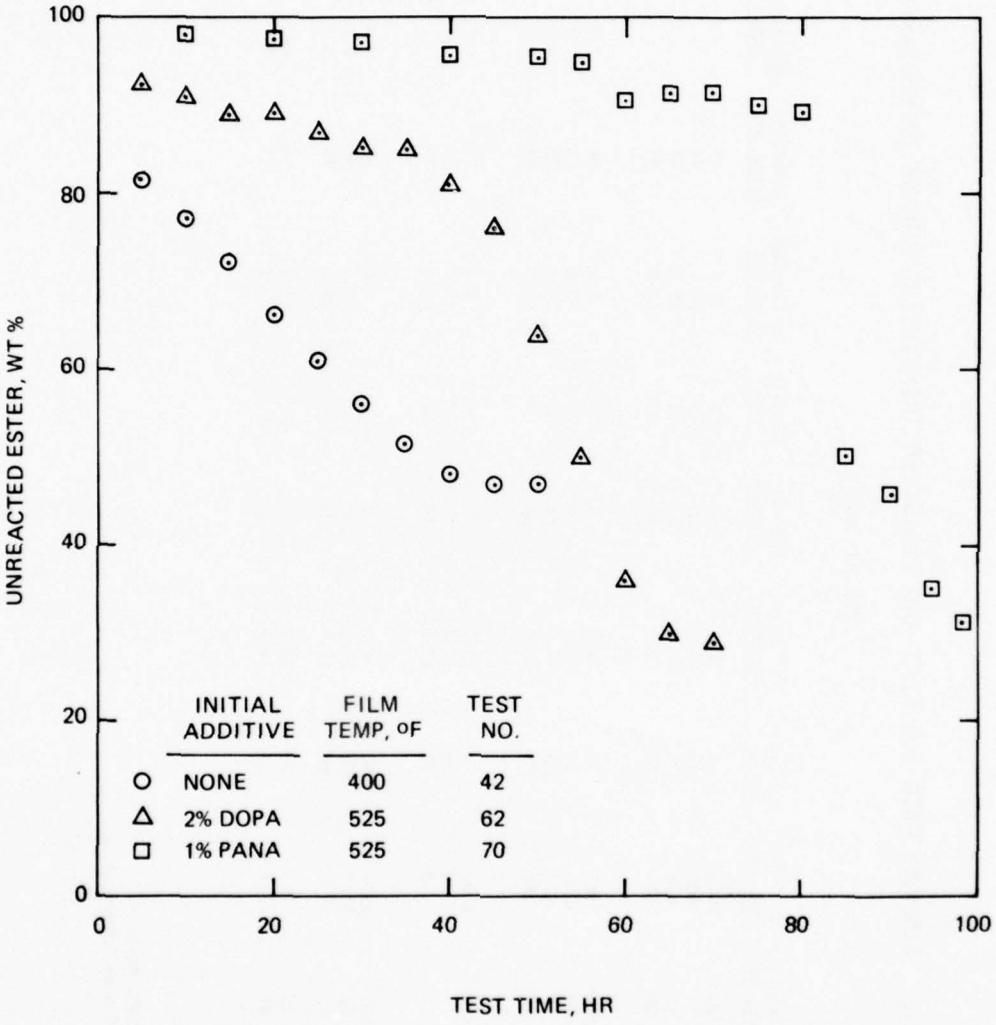


FIGURE 7. TMP ESTER CONSUMPTION TRENDS

consumption of the primary ester was noted during the period of additive effectiveness. Following complete additive depletion, rapid ester attack coincided with accelerated oxygen consumption rates and viscosity and acidity increases. The TMP test results differed somewhat in that, while still within the induction period, characteristic oxidation products at low concentration were evident by GC analysis. Subsequent to additive depletion, these products were generated at a faster rate but the major types were unchanged. These products were also unchanged from the types identified in previous work[1] with the neat polyol, viz., TMP diesters, GC residue, and, principally in the volatile condensate sample, heptanoic acid. These observations apply equally to the polyol blends containing the individual or combination additives listed in Table 5.

A measure of the relative effectiveness of the additives in TMP, and the induction period provided thereby, is given by a comparison of the cylinder rig residence times to complete additive depletion. These data are listed in Table 6. At the 525°F film temperature, the PANA additive was superior to DOPA by a factor of approximately two. Severe degradation and rapid additive losses at 550°F masked any performance distinction between the two inhibitors at this temperature. Data in Table 6 for the additive combination at two percent indicate that substitution of a portion of the DOPA material with PANA resulted in some protection of the former. The PANA additive was consumed at a slightly faster rate, comparing data for one percent PANA vs the two percent combination package, whereas the loss time for DOPA was extended. Data in this regard for the one percent combination were inconclusive because of interference in GC analysis for the DOPA additive by a minor degradation product. While thin layer chromatography techniques are available which would overcome this difficulty, the problem was only encountered near the conclusion of the program and there was not sufficient time for the acquisition of necessary materials.

Figure 8 presents data for inhibitor loss rate at a comparable 500°F film temperature. Results for the PANA material demonstrate a logarithmic function, as was shown with the DEHA basestock. The results for DOPA in Figure 8 indicate some fluctuation of data points from a logarithmic curve, but the deviation was not appreciable and may have been due to experimental variance. The plots in the figure for the two additives in combination also illustrate the protective effect of PANA relative to DOPA depletion. Throughout the test period covered, there was a consistent increase in the ratio of DOPA to PANA.

The influence of the combination additives on TMP deposition was unpredictable. As previously observed, the inhibited polyol ester showed no deposits at 500°F for either additive when used individually. Higher film temperatures (525° and 550°F) resulted in light sludge deposits, comparable to tests with the uninhibited TMP. Use of the two percent additive combination indicated a deleterious effect on deposits for this inhibitor package, as evidenced by the formation of carbon deposits at the higher temperatures (Table 5). In contrast, the one percent additive combination exhibited a significant beneficial effect on deposition. Light varnish deposits were formed at 525°F in duplicate tests with lubricant Z-1000. This deposit type is considered less harmful than all other types formed at the 525°F film

TABLE 6. COMPARISON OF ADDITIVE STABILITIES IN TMP

Film Temp, °F	Residence Time to Additive Depletion, sec			
	2% DOPA	1% PANA	2% (DOPA, PANA)	1% (DOPA, PANA)
500	>1274	>1266	>1530	
525	575	1309	1111, 627	*, 192 (avg)
550	159	157	212, 103	

* Indeterminable due to GC peak interference.

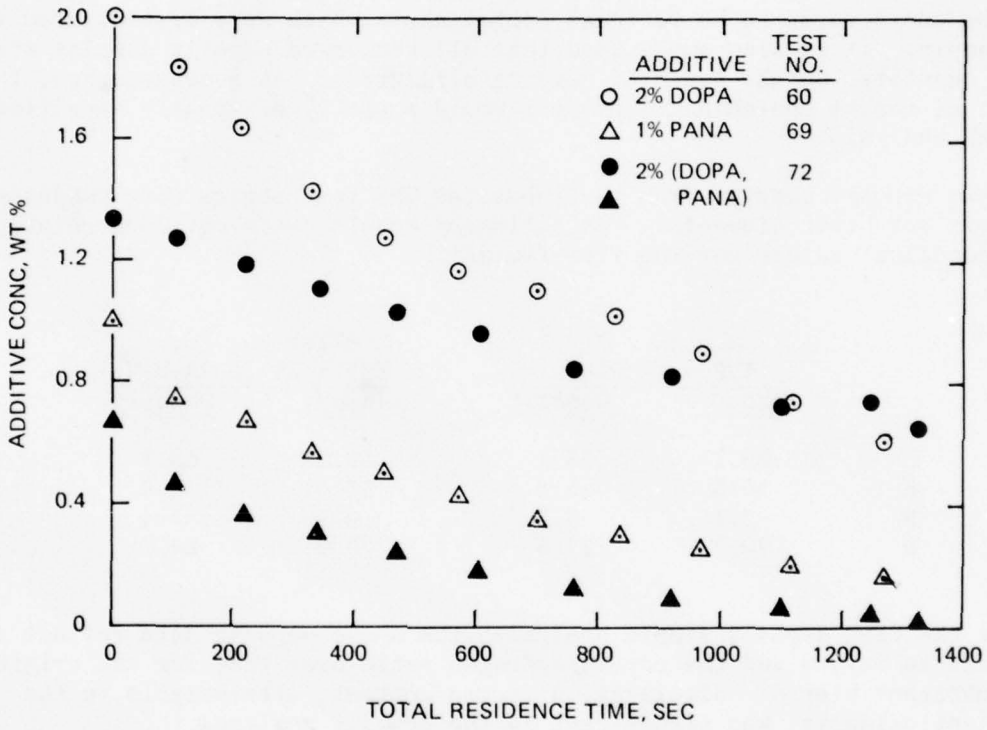


FIGURE 8. ADDITIVE DEPLETION IN TMP AT 500°F FILM TEMPERATURE

temperature. Thus, some additive interaction is indicated in the deposition process with TMP, and an optimum or synergistic blend of the DOPA and PANA antioxidants appears to exist.

Table 7 lists XRF analytical data for three TMP tests with one or both antioxidants present. Significant quantities of iron were found in fluid samples taken in the later stages of the tests. Once again, accelerated iron generation generally coincided with additive depletion, although this event was indeterminable for Test No. 78 due to DOPA peak interference in the GC analysis. A sufficient quantity of deposit sample for analysis was available only for Test No. 70. This sample showed extensive quantities of iron, primary, and silicon, sulphur, and calcium. The source of the last three elements can only be conjectured to be residual contaminants which were concentrated in the sample. It is also emphasized that all recovered deposit samples were of small quantity, on the order of several milligrams. As a consequence, the amount of random contaminants present would probably be greatly magnified in the XRF analysis.

Two deposit samples from the inhibited TMP test series were subjected to analysis for basic elements. The following results were obtained relative to theoretical values for the test fluids:

	Test No. 70, TMP + 1% PANA		Test No. 76, TMP + 2% (DOPA,PANA)	
	Theor.	Deposit	Theor.	Deposit
C	69.1	55.1	69.2	65.1
H	10.6	5.8	10.6	6.4
N	0.1	0.7	0.1	0.4
O	20.2	27.6	20.0	24.7

As for the DEHA deposit sample analysis, the above deposit data reflect an increase in oxygen and the carbon/hydrogen ratio over that for the original TMP lubricant blends. Similarly, nitrogen content, attributable to the amine antioxidants, was significant in the deposit analyses.

Mass spectroscopy was employed in further analysis of selected TMP deposit samples. These were samples from Test No. 70 (TMP + 1% PANA) and Test No. 76 (TMP + 2% DOPA, PANA). The derived spectrum for the former run is shown in Figure 9. The spectrum contains a multiplicity of fragment ions. While certain ions are characteristic of the polyol ester spectrum,[1] their abundances were minor. For example, m/e 113 was the base peak of the undegraded TMP ester, corresponding to the ester's C_7 acyl group. Figure 9 indicates a relative abundance of only some 15 percent for this ion. Other spectra similarities between the deposit sample and the original ester include the alkyl chain ions (m/e 43, 57, 71, and 85) and a trace abundance for m/e 341. The latter is postulated to be a fragment ion for the ester with

TABLE 7. RESULTS OF XRF ANALYSIS FOR SELECTED TMP TEST SAMPLES

Test No. 62, TMP + 2% DOPA	
0-45 hr	Nil
50	40 ppm Fe
55	90 ppm Fe
60	210 ppm Fe
65	1045 ppm Fe
70	2850 ppm Fe
Condensate	630 ppm Fe
Deposit	Insuff. sample

Test No. 70, TMP + 1% PANA	
0-90 hr	Nil
95	3325 ppm Fe
97	6080 ppm Fe
Condensate	37,600 ppm Fe
Deposit	7.3% Fe
	6.0% Si
	1.3% S
	3.2% Ca

Test No. 78, TMP + 1% (DOPA, PANA)	
0-30 hr	Nil
35	16 ppm Fe
40	40 ppm Fe
45	165 ppm Fe
Condensate	Insuff. sample
Deposit	Insuff. sample

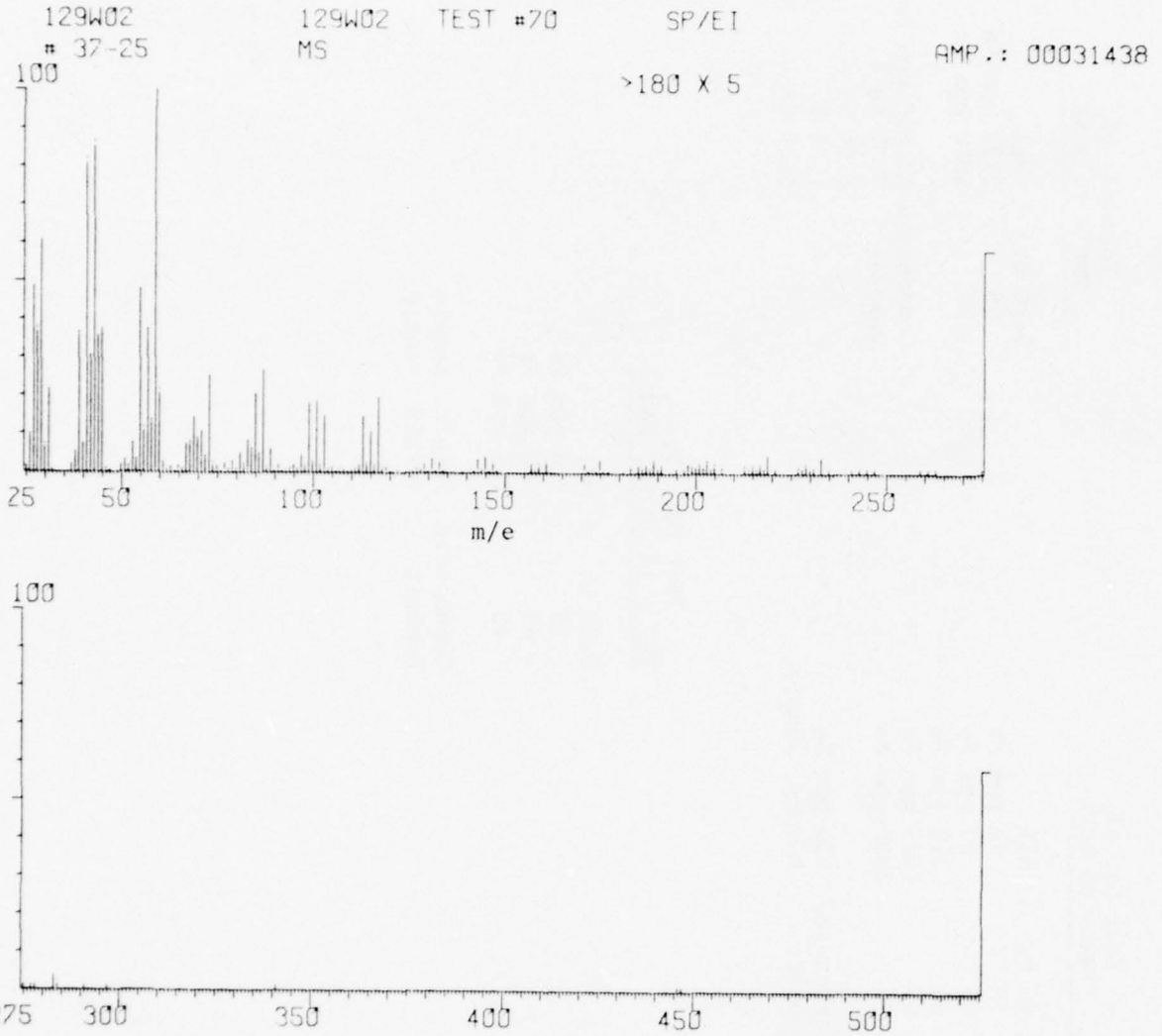
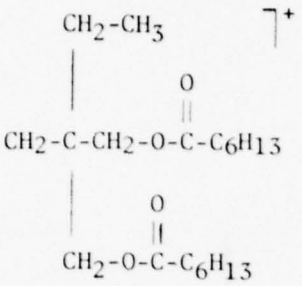


FIGURE 9. MASS SPECTRUM FOR TEST NO. 70 DEPOSIT SAMPLE

two acid side chains, of the structure



A distinctive ion series shown by the deposit sample was m/e 59, 73, 87,.... This series is typical for the chemical form $C_nH_{2n+1}NO$ and could partially account for the fate of the PANA nitrogen. The spectrum of Figure 9 also suggests evidence for the unsaturate ions of the form C_nH_{2n-1} and C_nH_{2n} by the peaks at m/e 27, 28, 41, 42, etc. There was no indication in the spectrum for the PANA aromatic rings, or that any degree of unsaturation shown by elemental analysis was attributable to an aromatic structure.

The Test No. 76 deposit spectrum is illustrated in Figure 10. In this case, the C_7 acyl ion at m/e 113 was prominent, being the base peak of the spectrum. Although the m/e 59 ion was low in abundance, the ion series for $C_nH_{2n+1}NO$ was again in evidence, as well as the unsaturate ion series of the type C_nH_{2n-1} and C_nH_{2n}. A weak response for the diester structure at m/e 341 was also noted. Once again, *no* ion patterns representative of amine additive structures were apparent for the Test No. 76 deposit.

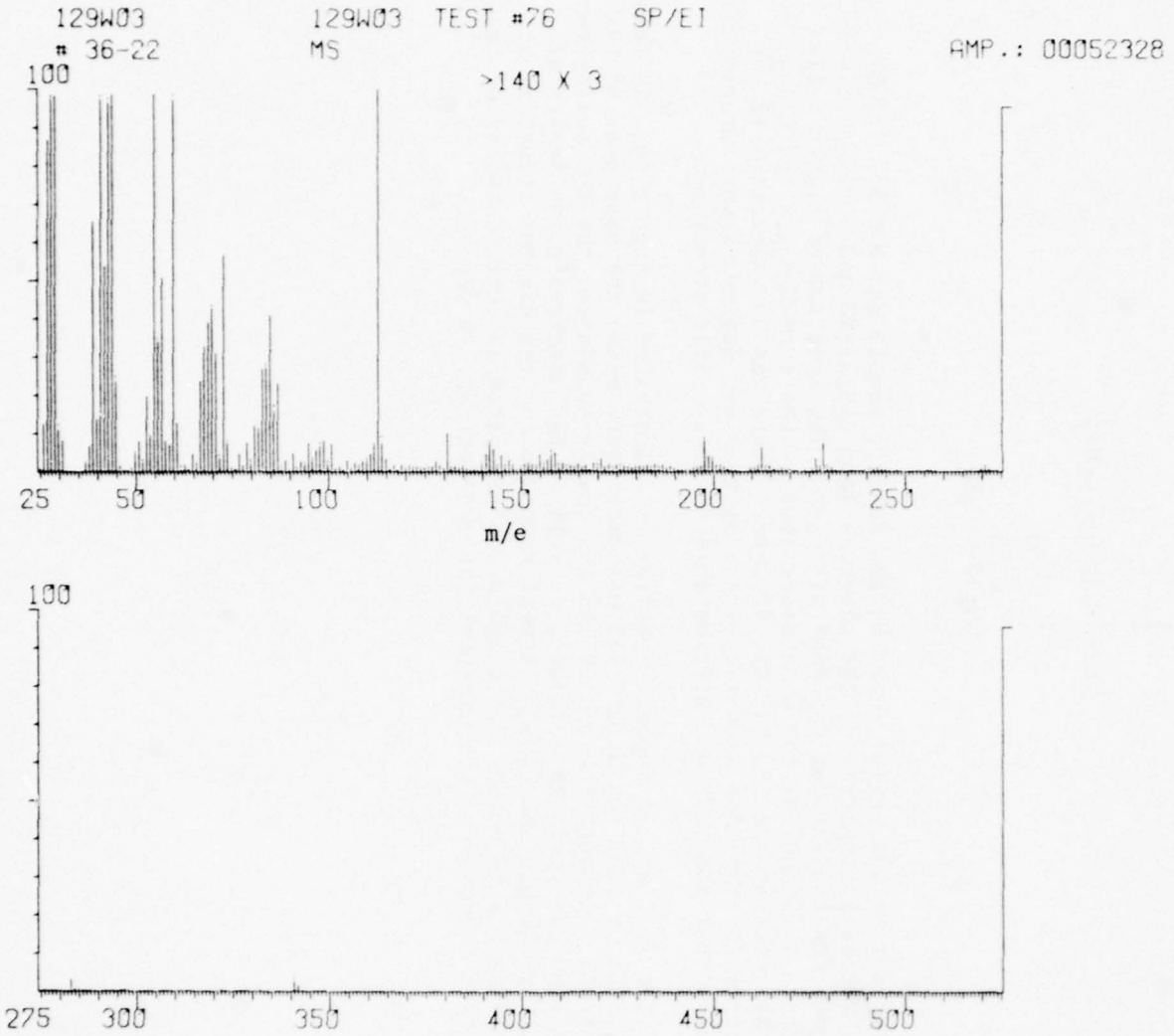


FIGURE 10. MASS SPECTRUM FOR TEST NO. 76 DEPOSIT SAMPLE

SECTION VII

CONCLUSIONS

The following conclusions are based on overall program findings employing the rotating cylinder rig as the experimental device:

- The principal mode of oxidative deterioration of the diester, di(2-ethylhexyl) adipate, was through loss of one or both of the ester alcohol groups. Products formed included several characteristic of the parent alcohol, mixed diesters and mono-esters, the half-acid ester, and, primarily, high-boiling components (GC residue), possibly polymeric or combination compounds of the diester fragments.

- The antioxidants, p,p'-dioctyldiphenylamine and phenyl-α-naphthylamine, effectively retarded oxidation of the diester prior to the time of complete additive depletion. At film temperatures equivalent to those which would severely degrade the uninhibited diester, there was no indication of a deleterious effect on deposits for the presence of either additive. At temperatures above 450°F, the PANA inhibitor apparently contributed to the formation of deposits of increased severity, as compared to DOPA.

- The mechanism of deposit formation by the inhibited diester is complex. Elemental analysis showed the composition of one deposit sample to be high in carbon/hydrogen ratio with some nitrogen. The deposit mass spectrum revealed several ions characteristic of the DEHA basestock. The deposit also contained significant amounts of inorganics, primarily iron. It is believed that this fact is critical to an understanding of the deposition mechanism. It is conjectured that a primary mechanism involves an interaction between nascent wearmetal particles and the ester degradation products, perhaps the half-acid ester and/or acyl group.

- Oxidative attack of the polyol ester, trimethylolpropane triheptanoate, was evidenced largely by cleavage of the C_7 acyl group and the formation of heptanoic acid, TMP diesters, and the high-boiling GC residue components.

- Employed individually, both of the amine antioxidants significantly extended the temperature capability of the TMP ester with respect to oxidative stability. Neither additive exhibited a tendency to promote deposit formation over that (light sludge) shown for the neat basestock. Using additive combinations in the ratio of 2:1 of DOPA:PANA, the two percent additive package did show a deleterious effect on deposits; whereas at one percent, a distinct beneficial effect was observed.

- As with the diester, analytical data for the polyol ester series strongly suggest that an interaction between the oxidation products and wearmetals, originating from a corrosive and/or wear process, is a significant factor in the deposition mechanism.

- Some comment is in order relative to the findings of this effort and field experience with aircraft turbine engine lubricants. Throughout this investigation, no deposits were formed except at experimental conditions resulting in severe ester degradation. For the inhibited esters, such degradation occurred only after additive depletion. In actual field use, bulk lubricant changes are negligible and total antioxidant depletion would not be expected—still engine deposits occur. This apparent anomaly is thought to be due to the fact that bulk lubricant changes do not reflect the condition of discrete portions of the lubricant within the engine. In areas of the engine receiving indirect lubrication or, perhaps, during conditions of high thermal soakback after engine shut-down, it is possible that rapid additive depletion and attendant ester breakdown and deposition occur for thin lubricant films within the engine.

SECTION VIII

RECOMMENDATIONS

The following recommendations resulting from this investigation are offered:

- To identify more precisely the chemical structure of both lubricant and deposit sample products, improved analytical techniques are required. It is believed that one technique worthy of indepth development for such application is mass spectroscopy employing chemical ionization.

- Additional study with respect to the influence of wearmetals on synthetic ester deposition is required for an adequate understanding of the deposition process. Definition of the type and source of active metal particles is desirable. The reactivity of other metal types found in an engine, such as copper and magnesium, is of interest. The deposit mechanism, or its negation, in a wear-free or nonmetallic environment should also be explored.

- To inhibit the wearmetal/lubricant product interaction for practical applications, the effect of lubricant additives such as metal deactivators and acid acceptors on the deposition process should be investigated.

- A beneficial synergistic effect on deposition was observed in this work with the combination antioxidant package at one percent. This finding suggests that a more thorough investigation of variations in additive concentrations could yield an additive package optimized with respect to deposit inhibition.

- Lastly, it is recommended that cylinder rig experiments with additional ester types of interest, including current specification-approved lubricants, be conducted to provide a more comprehensive delineation of synthetic lubricant performance/deposition characteristics.

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APPENDIX

ROTATING CYLINDER DEPOSITION TEST

SUMMARY DATA

TEST NO. 40

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-76-5

Test Conditions

Film temp, °F	350	Lubricant-in temp, °F	350
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.5	Test duration, hr	90
Total res. time, sec	1693.2		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.23	-	0.02	100	0
5	15.33	0.7	0.04	84.0	0.31
10	15.42	1.2	0.12	83.8	0.72
15	15.53	2.0	0.17	82.8	1.24
20	15.69	3.0	0.27	81.8	1.70
25	15.87	4.2	0.48	80.6	2.22
30	16.09	5.6	0.65	79.7	2.83
35	16.41	7.7	0.99	77.4	3.48
40	16.69	9.6	1.32	75.4	4.21
45	17.14	12.5	1.82	74.1	5.14
50	17.55	15.2	2.57	70.9	6.26
55	18.03	18.4	3.24	68.2	7.62
60	18.60	22.1	4.48	64.6	9.48
65	19.59	28.6	6.30	59.3	12.21
70	20.64	35.5	8.46	55.4	15.13
75	22.34	46.7	9.81	51.0	17.95
80	23.69	55.5	11.03	48.0	19.95
85	24.94	63.8	11.25	47.2	21.26
90	25.63	68.3	11.58	46.7	22.12

Oil loss, cm^3/hr 4.5

TEST NO. 41

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-76-5

Test Conditions

Film temp, °F	400	Lubricant-in temp, °F	400
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.1	Test duration, hr	50
Total res. time, sec	631.7		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.23	-	0.02	100	0
5	15.83	3.9	0.52	80.1	1.72
10	16.82	10.4	1.63	76.1	5.17
15	17.85	17.2	2.90	66.8	9.31
20	18.53	21.7	4.63	60.3	13.94
25	19.84	30.3	5.73	54.5	19.25
30	21.68	42.4	7.07	48.9	24.64
35	23.05	51.3	8.22	46.2	29.40
40	24.64	61.8	9.04	45.1	33.56
45	25.99	70.7	10.39	43.5	36.77
50	27.23	78.8	10.83	42.9	39.26

Oil loss, cm^3/hr 3.7

TEST NO. 42

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-76-5

Test Conditions

Film temp, °F	400	Lubricant-in temp, °F	400
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.1	Test duration, hr	50
Total res. time, sec	650.7		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	20	2
L sludge	80	48
	Overall Rating:	50

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	15.23	-	0.02	100	0
5	15.56	2.2	0.27	81.4	0.88
10	16.11	5.8	0.78	77.0	2.82
15	17.02	11.8	1.71	71.9	5.60
20	17.61	15.6	2.81	66.1	8.64
25	18.36	20.6	4.63	61.3	12.70
30	19.63	28.9	6.11	56.3	17.52
35	20.96	37.6	7.07	51.6	22.32
40	22.16	45.5	8.12	48.1	26.83
45	23.36	53.4	9.37	47.3	30.31
50	24.35	59.9	9.85	46.8	33.28

Oil loss, cm^3/hr 3.7

TEST NO. 43

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-76-5

Test Conditions

Film temp, °F	350	Lubricant-in temp, °F	350
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.5	Test duration, hr	100
Total res. time, sec	1800.2		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60
	Overall Rating:	60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	15.23	-	0.02	100	0
5	15.28	0.3	0.09	83.5	0.18
10	15.52	1.9	0.23	82.9	0.54
15	15.77	3.5	0.36	82.7	0.93
20	15.88	4.3	0.60	81.7	1.44
25	16.15	6.0	0.90	77.3	2.20
30	16.45	8.0	1.17	77.7	3.03
35	16.83	10.5	1.72	76.0	4.11
40	17.26	13.3	2.28	72.6	5.31
45	17.57	15.4	3.04	69.4	6.47
50	17.99	18.1	3.67	67.9	7.94
55	18.56	21.9	4.67	63.8	9.79
60	18.92	24.2	5.73	61.5	11.66
65	19.43	27.6	7.21	57.7	14.26
70	20.33	33.5	8.60	55.6	16.66
75	21.19	39.1	9.37	52.9	18.86
80	22.47	47.5	10.60	49.1	21.27
85	23.41	53.7	11.58	46.8	23.07
90	24.39	60.1	12.44	44.9	24.77
95	25.28	66.0	12.82	44.6	25.69
100	26.33	72.9	13.18	44.2	26.56

Oil loss, cm^3/hr 4.1

TEST NO. 44

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-77-1

Test Conditions

Film temp, °F	350	Lubricant-in temp, °F	350
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.5	Test duration, hr	50
Total res. time, sec	697.0		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.19	-	0.03	100	0
5	8.27	1.0	0.30	96.5	0.82
10	8.51	3.9	1.54	93.1	2.84
15	8.73	6.6	2.62	89.2	5.32
20	8.89	8.5	3.92	88.5	7.51
25	9.12	11.4	5.29	88.3	9.67
30	9.32	13.8	6.54	86.8	11.90
35	9.64	17.7	8.55	80.6	14.16
40	9.96	21.6	10.38	75.1	16.24
45	10.29	25.6	11.52	71.2	18.32
50	10.95	33.7	13.52	66.3	21.56

Oil loss, cm^3/hr 4.8

TEST NO. 45

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-77-1

Test Conditions

Film temp, °F	350	Lubricant-in temp, °F	350
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.5	Test duration, hr	50
Total res. time, sec	613.5		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.19	-	0.03	100	0
5	8.29	1.2	0.30	96.2	1.12
10	8.44	3.1	0.81	94.7	2.34
15	8.60	5.0	1.52	91.0	4.01
20	8.78	7.2	2.75	87.7	5.96
25	9.07	10.7	4.90	83.9	8.70
30	9.31	13.7	6.96	79.9	11.71
35	9.67	18.1	8.83	73.9	14.38
40	10.06	22.8	10.51	70.6	17.44
45	10.58	29.2	13.26	67.3	20.49
50	10.96	33.8	15.40	64.8	23.44

Oil loss, cm^3/hr 1.8

TEST NO. 53

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-77-1

Test Conditions

Film temp, °F	375	Lubricant-in temp, °F	375
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.3	Test duration, hr	35
Total res. time, sec	458.9		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.19	-	0.03	100	0
5	8.46	3.3	1.15	94.3	2.13
10	8.79	7.3	2.74	90.3	5.28
15	9.15	11.7	4.55	84.9	8.54
20	9.41	14.9	7.34	81.8	11.90
25	9.96	21.6	9.74	77.0	16.00
30	10.57	29.1	12.80	69.1	20.38
35	11.42	39.4	14.09	65.1	25.14

Oil loss, cm^3/hr 3.4

TEST NO. 54

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT O-77-1

Test Conditions

Film temp, °F	375	Lubricant-in temp, °F	375
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.3	Test duration, hr	35
Total res. time, sec	429.0		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.19	-	0.03	100	0
5	8.46	3.3	1.38	92.9	2.82
10	8.77	7.1	3.13	90.2	6.40
15	9.16	11.8	5.05	85.6	10.05
20	9.56	16.7	7.06	81.9	14.23
25	10.07	23.0	9.57	75.9	18.74
30	10.71	30.8	11.46	71.1	23.44
35	11.45	39.8	13.35	65.7	28.04

Oil loss, cm^3/hr 4.3

TEST NO. 55

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1004

Test Conditions

Film temp, °F	350	Lubricant-in temp, °F	350
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.5	Test duration, hr	135
Total res. time, sec	2935.0		

Deposit Rating

Deposit Type	Area, %	Demerits
Clean	100	0

Overall Rating: 0

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.65	-	0.05	100	0
5	8.66	0.1	0.08	-	0.11
10	8.67	0.2	0.08	100	0.19
15	8.65	0.0	0.08	-	0.24
20	8.70	0.6	0.00	100	0.26
25	8.70	0.6	0.04	-	0.28
30	8.67	0.2	0.08	100	0.75
35	8.65	0.0	0.05	-	1.05
40	8.65	0.0	0.08	99.8	1.36
45	8.70	0.6	0.06	-	1.63
50	8.70	0.6	0.08	99.7	1.91
55	8.71	0.7	0.10	-	2.17
60	8.69	0.5	0.10	99.3	2.31
65	8.71	0.7	0.10	-	2.31
70	8.72	0.8	0.10	99.4	2.32
75	8.70	0.6	0.10	-	2.53
80	8.72	0.8	0.11	99.0	2.80
85	8.73	0.9	0.13	-	3.07
90	8.73	0.9	0.13	97.9	3.10
95	8.73	0.9	0.15	-	3.16
100	8.74	1.0	0.15	98.2	3.20

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt%	Oxygen Consumption, ℓ at OPT
105	8.74	1.0	0.17	-	3.32
110	8.75	1.2	0.18	98.3	3.41
115	8.77	1.4	0.20	-	3.48
120	8.75	1.2	0.21	98.2	3.61
125	8.76	1.3	0.24	-	3.70
130	8.77	1.4	0.26	98.1	3.84
135	8.78	1.5	0.28	98.2	4.09

Oil loss, cm^3/hr 3.9

TEST NO. 56

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1004

Test Conditions

Film temp, °F	400	Lubricant-in temp, °F	400
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	18.1	Test duration, hr	100
Total res. time, sec	1411.1		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.65	-	0.05	100	0
5	8.69	0.5	0.03	-	0.26
10	8.70	0.6	0.06	96.9	0.58
15	8.72	0.8	0.14	-	0.88
20	8.72	0.8	0.18	96.5	1.18
25	8.74	1.0	0.22	-	1.44
30	8.78	1.5	0.24	96.2	1.74
35	8.79	1.6	0.28	-	2.04
40	8.80	1.7	0.35	96.2	2.30
45	8.83	2.1	0.40	-	2.58
50	8.86	2.4	0.45	96.1	2.87
55	8.87	2.5	0.51	-	3.11
60	8.89	2.8	0.60	95.7	3.39
65	8.92	3.1	0.66	-	3.65
70	8.94	3.4	0.76	95.6	4.02
75	8.97	3.7	0.82	-	4.38
80	9.00	4.0	0.92	95.5	4.71
85	9.00	4.0	1.02	-	5.00
90	9.04	4.5	1.07	94.7	5.34
95	9.06	4.7	1.13	-	5.77
100	9.07	4.9	1.24	94.1	6.27

Oil loss, cm^3/hr 1.8

TEST NO. 57

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1004

Test Conditions

Film temp, °F	450	Lubricant-in temp, °F	450
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.8	Test duration, hr	95
Total res. time, sec	1813.8		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.65	-	0.05	100	0
5	8.71	0.7	0.20	-	0.60
10	8.78	1.5	0.49	95.3	1.26
15	8.81	1.8	0.57	-	3.04
20	8.85	2.3	0.84	95.1	4.62
25	8.90	2.9	1.09	-	5.64
30	8.93	3.2	1.35	95.3	5.90
35	9.00	4.0	1.61	-	7.55
40	9.04	4.5	1.94	94.2	9.01
45	9.05	4.6	2.21	-	9.82
50	9.14	5.7	2.58	94.1	10.52
55	9.24	6.8	2.94	93.5	11.13
60	9.27	7.2	3.33	93.3	11.94
65	9.30	7.5	3.62	93.6	12.79
70	9.41	8.8	4.07	92.9	13.44
75	9.42	8.9	4.52	92.8	14.12
80	9.69	12.0	6.21	87.6	15.08
85	11.67	34.9	13.80	71.4	22.71
90	15.07	74.2	24.66	55.0	32.56
95	19.84	129.4	35.48	50.1	39.82

Oil loss, cm^3/hr 4.9

TEST NO. 58

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1004

Test Conditions

Film temp, °F	475	Lubricant-in temp, °F	475
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.6	Test duration, hr	50
Total res. time, sec	674.5		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	8.65	-	0.05	100	0
5	8.75	1.2	0.43	98.8	1.17
10	8.83	2.1	0.90	96.6	2.90
15	8.95	3.5	1.44	95.7	4.67
20	9.00	4.0	1.99	95.0	5.96
25	9.08	5.0	2.71	93.2	7.52
30	9.39	8.6	3.95	91.7	10.62
35	10.20	17.9	7.95	89.3	18.02
40	12.17	40.7	14.36	69.2	30.01
45	14.76	70.6	23.15	54.2	44.44
50	19.26	122.7	29.53	48.5	56.00

Oil loss, cm^3/hr 7.3

TEST NO. 59

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1006

Test Conditions

Film temp, °F	450	Lubricant-in temp, °F	450
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.8	Test duration, hr	100
Total res. time, sec	1302.9		

Deposit Rating

Deposit Type	Area, %	Demerits
Clean	100	0

Overall Rating: 0

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	16.16	-	0.03	100	0
5	16.33	1.1	0.06	-	0.43
10	16.40	1.5	0.08	97.7	0.84
15	16.49	2.0	0.11	-	1.34
20	16.55	2.4	0.13	97.3	1.73
25	16.58	2.6	0.15	-	2.20
30	16.71	3.4	0.22	97.3	2.70
35	16.71	3.4	0.25	-	3.24
40	16.77	3.8	0.27	97.3	3.84
45	16.81	4.0	0.31	-	4.14
50	16.90	4.6	0.32	97.3	4.66
55	16.96	5.0	0.36	97.3	4.89
60	17.00	5.2	0.42	97.1	5.31
65	17.11	5.9	0.45	97.0	5.76
70	17.16	6.2	0.51	96.8	5.94
75	17.21	6.5	0.53	96.8	6.17
80	17.21	6.5	0.60	96.5	6.54
85	17.31	7.1	0.68	95.7	6.86
90	17.43	7.9	0.69	94.8	7.26
95	17.49	8.2	0.72	93.6	7.56
100	17.52	8.4	0.76	93.7	8.00

Oil loss, cm^3/hr 1.3

TEST NO. 60

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1006

Test Conditions

Film temp, °F	500	Lubricant-in temp, °F	500
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.4	Test duration, hr	100
Total res. time, sec	1273.5		

Deposit Rating

Deposit Type	Area, %	Demerits
Clean	100	0

Overall Rating: 0

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	16.16	-	0.03	100	0
5	16.43	1.7	0.07	-	0.49
10	16.64	3.0	0.20	99.0	1.32
15	16.82	4.1	0.29	-	2.30
20	16.90	4.6	0.35	97.8	3.08
25	17.02	5.3	0.47	-	3.99
30	17.22	6.6	0.53	95.6	4.61
35	17.34	7.3	0.60	-	4.90
40	17.49	8.2	0.67	94.3	5.24
45	17.61	9.0	0.74	-	5.65
50	17.69	9.5	0.83	93.7	6.04
55	17.78	10.0	0.85	93.0	6.36
60	17.86	10.5	0.94	92.6	6.53
65	18.01	11.4	0.98	92.2	6.97
70	18.13	12.2	1.05	93.8	7.60
75	18.20	12.6	1.11	93.4	8.25
80	18.32	13.4	1.21	93.1	8.88
85	18.34	13.5	1.28	92.8	9.60
90	18.51	14.5	1.39	91.3	9.99
95	18.64	15.3	1.41	89.1	10.33
100	18.78	16.2	1.43	87.3	10.84

Oil loss, cm^3/hr 1.5

TEST NO. 61

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1006

Test Conditions

Film temp, °F	550	Lubricant-in temp, °F	550
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.1	Test duration, hr	25
Total res. time, sec	280.5		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	16.16	-	0.03	100	0
5	18.21	12.7	2.35	86.0	8.43
10	21.11	30.6	5.61	71.1	22.48
15	26.23	62.3	9.09	48.3	43.34
20	34.25	111.9	14.07	33.4	70.56
25	46.46	187.5	15.96	29.0	87.47

Oil loss, cm^3/hr 7.8

TEST NO. 62

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1006

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, in, X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	70
Total res. time, sec	871.4		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	16.16	-	0.03	100	0
5	16.61	2.8	0.31	92.3	1.58
10	17.33	7.2	0.76	91.3	3.54
15	17.72	9.6	1.07	89.0	5.72
20	17.98	11.3	1.32	88.6	7.13
25	18.34	13.5	1.61	86.9	8.78
30	18.71	15.8	1.86	84.7	10.52
35	19.02	17.7	2.13	84.6	11.82
40	19.56	21.0	2.71	81.3	14.14
45	20.59	27.4	3.47	75.6	17.66
50	22.76	40.8	5.60	64.1	24.76
55	27.51	70.2	7.85	49.9	38.02
60	36.60	126.5	11.12	35.8	56.48
65	51.67	219.7	13.67	30.4	68.92
70	66.51	311.6	14.76	29.3	79.39

Oil loss, cm^3/hr 3.9

TEST NO. 63

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1007

Test Conditions

Film temp, °F	450	Lubricant-in temp, °F	450
Film thickness, in, X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.8	Test duration, hr	100
Total res. time, sec	1447.2		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	95	9.5
L sludge	5	3

Overall Rating: 12.5

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.34	-	0.03	100	0
5	8.36	0.2	0.06	-	0.71
10	8.37	0.4	0.15	99.1	1.78
15	8.41	0.8	0.20	-	3.20
20	8.43	1.1	0.29	98.9	3.59
25	8.46	1.4	0.38	-	4.02
30	8.49	1.8	0.49	98.9	4.41
35	8.50	1.9	0.59	-	4.62
40	8.52	2.2	0.72	97.4	4.76
45	8.57	2.8	0.83	-	4.96
50	8.58	2.9	0.94	97.8	5.06
55	8.63	3.5	1.07	97.3	5.29
60	8.63	3.5	1.16	97.0	5.71
65	8.66	3.8	1.25	97.1	5.98
70	8.69	4.2	1.39	97.1	6.07
75	8.69	4.2	1.52	97.7	6.37
80	8.73	4.7	1.64	97.9	6.43
85	8.74	4.8	1.77	97.9	6.70
90	8.78	5.3	1.95	97.8	7.25
95	8.78	5.3	2.08	96.6	7.35
100	8.82	5.8	2.23	96.1	7.40

Oil loss, cm^3/hr 2.6

TEST NO. 64

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1007

Test Conditions

Film temp, °F	475	Lubricant-in temp, °F	475
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.6	Test duration, hr	100
Total res. time, sec	1478.5		

Deposit Rating

Deposit Type	Area, %	Demerits
M varnish	100	30

Overall Rating: 30

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	8.34	-	0.03	100	0
5	8.35	0.1	0.10	-	0.24
10	8.42	1.0	0.23	98.1	0.65
15	8.45	1.3	0.33	-	1.36
20	8.47	1.6	0.45	98.1	1.44
25	8.52	2.2	0.60	-	1.70
30	8.53	2.3	0.76	96.9	2.17
35	8.56	2.6	0.85	-	2.36
40	8.59	3.0	1.03	96.8	2.78
45	8.62	3.4	1.20	-	3.35
50	8.67	4.0	1.32	96.2	3.64
55	8.72	4.6	1.43	96.2	4.08
60	8.73	4.7	1.61	96.2	4.19
65	8.74	4.8	1.77	96.1	4.73
70	8.77	5.2	1.92	95.8	5.44
75	8.80	5.5	2.05	95.5	5.68
80	8.82	5.8	2.26	95.6	6.42
85	8.84	6.0	2.37	95.9	7.35
90	8.87	6.4	2.50	96.0	7.54
95	8.90	6.7	2.80	95.5	8.12
100	9.00	7.9	3.12	94.1	9.14

Oil loss, cm^3/hr 3.4

TEST NO. 65

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1007

Test Conditions

Film temp, °F	500	Lubricant-in temp, °F	500
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.4	Test duration, hr	100
Total res. time, sec	1451.4		

Deposit Rating

Deposit Type	Area, %	Demerits
H varnish	100	50

Overall Rating: 50

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	8.34	-	0.03	100	0
5	8.43	1.1	0.17	-	0.32
10	8.44	1.2	0.31	97.1	0.84
15	8.47	1.6	0.46	-	1.26
20	8.50	1.9	0.62	96.9	1.66
25	8.53	2.3	0.78	-	2.01
30	8.57	2.8	0.96	96.3	2.44
35	8.60	3.1	1.13	-	2.87
40	8.64	3.6	1.34	95.7	3.30
45	8.68	4.1	1.48	-	3.84
50	8.71	4.4	1.61	95.2	4.24
55	8.75	4.9	1.81	95.1	4.61
60	8.79	5.4	2.03	95.1	4.86
65	8.80	5.5	2.22	94.9	5.08
70	8.83	5.9	2.41	95.1	5.40
75	8.87	6.4	2.55	95.1	5.70
80	8.89	6.6	2.72	95.2	5.94
85	8.93	7.1	3.04	95.2	6.34
90	8.97	7.6	3.34	94.2	6.59
95	9.00	7.9	3.59	94.1	7.00
100	9.03	8.3	3.66	94.1	7.37

Oil loss, cm^3/hr 2.8

TEST NO. 67

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1007

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100, moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	45
Total res. time, sec	583.5		

Deposit Rating

Deposit Type	Area, %	Demerits
M crinkled carbon	100	130

Overall Rating: 130

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	8.34	-	0.03	100	0
5	8.45	1.3	0.44	96.2	1.29
10	8.52	2.2	0.93	96.8	2.49
15	8.58	2.9	1.39	94.2	3.60
20	8.66	3.8	1.81	94.2	4.84
25	8.74	4.8	2.33	94.3	6.20
30	8.92	7.0	3.74	92.7	8.72
35	9.63	15.5	7.50	83.1	15.24
40	12.37	48.3	15.91	67.3	32.51
45	21.35	156.0	22.45	50.8	46.14

Oil loss, cm^3/hr 11.1

TEST NO. 69

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1010

Test Conditions

Film temp, °F	500	Lubricant-in temp, °F	500
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100, moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.4	Test duration, hr	100
Total res. time, sec	1266.4		

Deposit Rating

Deposit Type	Area, %	Demerits
Clean	100	0

Overall Rating: 0

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.49	-	0.02	100	0
5	15.66	1.1	0.04	-	0.06
10	15.73	1.5	0.09	99.1	0.38
15	15.80	2.0	0.11	-	0.75
20	15.85	2.3	0.13	98.9	0.80
25	15.90	2.6	0.19	-	1.08
30	15.96	2.8	0.22	98.7	1.54
35	16.06	3.7	0.25	-	1.56
40	16.07	3.7	0.32	98.5	1.57
45	16.11	4.0	0.36	-	1.60
50	16.17	4.4	0.41	98.4	1.80
55	16.24	4.8	0.43	98.0	1.98
60	16.28	5.1	0.49	96.9	2.05
65	16.34	5.5	0.56	96.3	2.28
70	16.40	5.9	0.61	95.5	2.41
75	16.49	6.5	0.65	94.9	2.62
80	16.51	6.6	0.70	94.4	2.87
85	16.55	6.8	0.76	94.2	3.32
90	16.65	7.5	0.81	93.8	3.74
95	16.73	8.0	0.85	93.2	4.03
100	16.82	8.6	0.88	92.5	4.19

Oil loss, cm^3/hr 1.2

TEST NO. 70

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1010

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	97
Total res. time, sec	1976.4		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	15.49	-	0.02	100	0
5	15.73	1.5	0.09	-	0.05
10	15.82	2.1	0.18	98.3	0.52
15	15.90	2.6	0.27	-	0.66
20	16.07	3.7	0.34	97.7	1.06
25	16.17	4.4	0.43	-	1.39
30	16.23	4.8	0.50	97.0	1.70
35	16.37	5.7	0.61	-	2.01
40	16.50	6.5	0.72	95.5	2.10
45	16.53	6.7	0.86	-	2.10
50	16.71	7.9	1.01	95.6	2.39
55	16.85	8.8	1.10	95.3	2.82
60	16.96	9.5	1.24	90.5	3.14
65	17.12	10.5	1.31	90.5	3.60
70	17.28	11.6	1.40	91.5	4.04
75	17.41	12.4	1.48	90.2	4.21
80	17.68	14.1	1.70	89.4	4.56
85	17.97	16.0	2.15	50.0	5.25
90	22.05	42.3	6.50	45.8	10.97
95	40.63	162	14.70	35.2	17.22
97*	92.00	494	15.94	31.4	19.08

Oil loss, cm^3/hr 6.2

*Test terminated at 97 hr due to oil thickening and pumping difficulties.

TEST NO. 71

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1010

Test Conditions

Film temp, °F	550	Lubricant-in temp, °F	550
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.1	Test duration, hr	25
Total res. time, sec	269.1		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	100	60

Overall Rating: 60

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	15.49	-	0.02	100	0.0
5	16.22	4.7	0.43	97.8	2.63
10	17.20	11.0	1.55	89.1	5.94
15	19.21	24.0	4.98	72.2	16.76
20	24.60	58.8	7.62	50.9	37.53
25	30.71	98.3	12.46	40.4	62.61

Oil loss, cm^3/hr 3.4

TEST NO. 72

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1011

Test Conditions

Film temp, °F	500	Lubricant-in temp, °F	500
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.4	Test duration, hr	100
Total res. time, sec	1530.2		

Deposit Rating

Deposit Type	Area, %	Demerits
Clean	100	0

Overall Rating: 0

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.88	-	0.03	100	0
5	16.14	1.6	0.04	-	0.34
10	16.31	2.7	0.05	99.4	0.74
15	16.45	3.6	0.09	-	1.18
20	16.52	4.0	0.12	97.4	1.31
25	16.59	4.5	0.18	-	1.60
30	16.70	5.2	0.25	97.1	1.91
35	16.77	5.6	0.31	-	2.14
40	16.80	5.8	0.39	96.7	2.25
45	16.93	6.6	0.43	-	2.62
50	17.01	7.1	0.51	96.5	2.70
55	17.10	7.7	0.59	96.4	3.22
60	17.22	8.4	0.61	96.2	3.47
65	17.29	8.9	0.68	95.8	3.66
70	17.45	9.9	0.73	94.6	4.10
75	17.51	10.3	0.80	95.5	4.36
80	17.63	11.0	0.85	96.0	4.41
85	17.78	12.0	0.92	94.4	4.55
90	17.82	12.2	1.02	94.9	4.87
95	17.78	12.0	1.04	92.3	5.26
100	17.81	12.2	1.05	93.0	5.57

Oil loss, cm^3/hr 3.8

TEST NO. 76

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1011

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, $in_3 \times 10^{-3}$	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	95
Total res. time, sec	1190.9		

Deposit Rating

Deposit Type	Area, %	Demerits
L smooth carbon	50	45
L crinkled carbon	50	60

Overall Rating: 105

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.88	-	0.03	100	0
5	16.28	2.5	0.08	-	1.12
10	16.45	3.6	0.19	98.8	2.08
15	16.60	4.5	0.30	-	2.77
20	16.71	5.2	0.43	96.2	3.39
25	16.88	6.3	0.58	-	3.90
30	17.02	7.2	0.75	96.3	4.38
35	17.12	7.8	0.91	-	4.79
40	17.29	8.9	1.07	94.9	5.48
45	17.45	9.9	1.18	-	6.44
50	17.57	10.6	1.23	94.9	7.36
55	17.49	10.1	2.37	93.5	9.02
60	17.91	12.8	1.66	92.3	9.82
65	18.50	16.5	2.17	89.5	11.81
70	19.24	21.2	3.17	83.8	13.96
75	19.35	21.9	3.55	83.9	15.14
80	19.71	24.1	3.83	84.0	17.16
85	20.76	30.7	5.76	77.9	22.54
90	24.00	51.1	8.68	56.9	35.86
95	27.26	71.7	20.25	49.8	45.58

Oil loss, cm^3/hr 2.0

TEST NO. 77

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Y-1011

Test Conditions

Film temp, °F	550	Lubricant-in temp, °F	550
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.1	Test duration, hr	25
Total res. time, sec	278.7		

Deposit Rating

Deposit Type	Area, %	Demerits
L sludge	50	30
L crinkled carbon	50	60

Overall Rating: 90

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.88	-	0.03	100	0
5	17.31	9.0	1.60	92.5	5.07
10	18.20	14.6	2.80	86.6	10.51
15	19.91	25.4	5.65	76.1	17.37
20	23.39	47.3	9.34	59.1	29.52
25	30.06	89.3	12.86	49.3	50.11

Oil loss, cm^3/hr 10.6

TEST NO. 78

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Z-1000

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, in. X 10^{-3}	4	Airflow, liters/hr	100,moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	45
Total res. time, sec	515.0		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, ℓ at OPT
0	15.66	-	0.03	100	0
5	16.03	2.4	0.26	100	0.68
10	16.23	3.6	0.48	99.5	1.35
15	16.46	5.1	0.66	97.9	1.99
20	16.78	7.2	0.90	95.8	2.68
25	17.18	9.7	1.34	94.1	4.14
30	17.51	11.8	1.91	90.6	5.42
35	19.58	25.0	4.02	78.8	11.22
40	22.01	40.5	7.24	62.1	20.48
45	31.39	100.4	14.08	36.4	37.64

Oil loss, cm^3/hr 3.1

TEST NO. 79

OXIDATIVE
ROTATING CYLINDER DEPOSITION TEST SUMMARY DATA
ON LUBRICANT Z-1000

Test Conditions

Film temp, °F	525	Lubricant-in temp, °F	525
Film thickness, in, X 10^{-3}	4	Airflow, liters/hr	100, moist
Lubricant flow, cm^3/min	10	Lubricant charge, cm^3	1000
Average res. time, sec/cycle	17.3	Test duration, hr	25
Total res. time, sec	312.4		

Deposit Rating

Deposit Type	Area, %	Demerits
L varnish	100	10

Overall Rating: 10

Test Lubricant Performance

Test Time,hr	100°F Vis,cs	100°F Vis Increase,%	NN Change, mg KOH/g	Unreacted Ester,wt %	Oxygen Consumption, % at OPT
0	15.66	-	0.03	100	0
5	16.06	2.6	0.25	97.7	0.88
10	17.05	8.9	1.56	91.3	3.71
15	19.27	23.1	3.74	81.8	11.00
20	23.84	52.2	7.68	55.5	24.19
25	33.51	114.0	14.85	35.3	45.01

Oil loss, cm^3/hr 10.4

*U.S.Government Printing Office: 1979 — 657-002/729